Toward a definition of blueprint of virgin olive oil by comprehensive two-dimensional gas chromatography

TOWARD A DEFINITION OF BLUEPRINT OF VIRGIN OLIVE OIL BY COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Abstract

This study investigates the applicability of an iterative approach aimed at defining a chemical blueprint of virgin olive oil volatiles to be correlated to the product sensory quality. The investigation strategy proposed allows to fully exploit the informative content of a comprehensive multidimensional gas chromatography (GC × GC) coupled to a mass spectrometry (MS) data set. Olive oil samples (19), including 5 reference standards, obtained from the International Olive Oil Council, and commercial samples, were submitted to a sensory evaluation by a Panel test, before being analysed in two laboratories using different instrumentation, column set, and software elaboration packages in view of a cross-validation of the entire methodology. A first classification of samples based on untargeted *peak features* information, was obtained on raw data from two different column combinations (apolar × polar and polar × apolar) by applying unsupervised Multivariate Analysis (i.e., Principal Component Analysis - PCA). However, to improve effectiveness and specificity of this classification, *peak features* were reliably identified (261 compounds), on the basis of the MS spectrum and linear retention index matching, and subjected to successive pair-wise comparisons based on 2D patterns, which revealed peculiar distribution of chemicals correlated with samples sensory classification. The most informative compounds were thus identified and collected in a "blueprint" of specific defects (or combination of defects) successively adopted to discriminate Extra Virgin from defected oils (i.e. *lampante* oil) with the aid of a supervised approach, i.e., Partial Least Squares-Discriminant Analysis (PLS-DA). In this last step, the principles of sensomics, which assigns higher information potential to analytes with lower odor threshold proved to be successful, and a much more powerful discrimination of samples was obtained in view of a sensory quality assessment.

Keywords: olive oil; sensomics; volatile compounds; comprehensive multidimensional gas chromatography (GCxGC); fingerprinting; pattern recognition; aroma defects

1. Introduction

Modern -omics disciplines investigate sample constituents considered collectively (primary and secondary metabolites, compounds generated by thermal treatments and/or enzymatic activity) [1,2] and represent the route of choice for a comprehensive evaluation of food attributes, in particular if sensory properties (sensomics and flavoromics) are unequivocally correlated to a specific distribution of chemicals [3-5].

Sensomics focuses analytical efforts on revealing sensorially-active compounds (odorants and tastants) in order to link the chemical composition of a food with its sensory quality (flavor) [6]. From this perspective, the analytical process through which the peculiar product profile (i.e. the blueprint) is revealed closely fits the aims of the above mentioned - omics disciplines, in addition to comprehensive information on chemical composition, quality and authenticity,.

However, the number of volatiles effectively contributing to food aroma is relatively small, and the detection, identification, and quantitation of odor-active components, sometimes occurring at trace (ng/kg) levels, require complex procedures that combine effective extraction, powerful separation and highly informative (identity confirmation and mass quantitation) detection.

In this perspective, two-dimensional comprehensive gas chromatography (GC×GC) coupled to mass spectrometry (MS) can be successful in revealing the sensory-related chemical blueprint [7] of high quality foods since it fully exploits the informative potential of each analytical dimension (separation and detection) by (a) increasing the separation power, (b) ordering analytes through logic structures over the chromatographic plane and (c) by enhancing detection sensitivity,. In particular, the possibility of extending the analytical investigation to a broader range of chemicals, if compared to mono-dimensional approaches, improves fingerprinting sensitivity and effectiveness of sample classification [8,9].

Methods for reliable selections of 2D peak information and alignment across set of chromatograms were recently reviewed by Reichenbach *et al.* [10]. In particular, the most performing approaches are those referred to as *peak feature* methods, i.e. methods that enable not only, untargeted analytes alignment (based on peak features, i.e., retention times and MS fragmentation pattern similarity) but also pattern recognition and pair-wise comparisons based on 2D peaks distribution over the chromatographic plane.

In this study, the informative potentials of GC×GC-MS in revealing the chemical blueprint of (Extra) Virgin Olive Oil (EVO and VO) have been investigated by adopting advanced fingerprinting methods for reliable peak selection and focusing on peculiar distributions of sensory-active volatiles (aroma compounds) related to quality attributes currently regulated by international organisms (European Union (EU), International Olive Council (IOC), and the Codex Alimentarius) [11-14].

Interestingly, olive oil is presently the only food product whose sensory attributes are officially regulated, and sensory assessment carried out through well-standardized protocols [11-14] by highly and continuously (permanently) trained panelists. On the basis of the presence (or absence) and the intensities of specific

84 defects and the intensity of the “fruity” perception, evaluated by smelling and tasting, Virgin Olive Oil can
85 be classified into three categories (determining its economic value) namely EVO, VO, and *lampante* oil [15].
86 Undoubtedly, volatile compounds play a crucial role in defining olive oil sensory quality and strong efforts
87 have been dedicated to unravel the composition of this informative fraction, to understand correlations
88 with quality attributes [16-18]. The relative distribution of volatiles depends on several parameters (i.e.
89 cultivar, geographical origin, fruit ripeness, processing practices, and storage [18-27]) and the identification
90 of an unequivocal fingerprint correlated to quality may be a difficult task. Most of these variables concur to
91 determine the intensity and quality of the green and fruity perception, while the presence of defects is
92 mainly due to inappropriate manufacturing practices. Olive storage has to last no longer than 24-48h under
93 appropriate conditions to avoid the formation of three main defects, namely mustiness or mold, winey or
94 vinegary, and fusty. Such defects are mainly due to yeast and fungi metabolic activity and aerobic or
95 anaerobic fermentations, respectively [13]. While inappropriate oil storage conditions after production may
96 lead to muddy and rancid defects, the former may be originated by an excessive contact of the oil with
97 sediment in tanks, and the latter by an auto-oxidation process. The descriptor of the fusty and muddy
98 defects was recently unified due to several misinterpretations [12], although the origin of such defects is
99 very different both from the technological and chemical viewpoint.
100 In the panorama of existing studies, just few of them focused on the identification of markers of the
101 specific defects by adopting reference standard samples provided by the IOC [27, 28]. Morales and co-
102 workers [26], developed a dynamic headspace (D-HS) sampling procedure to extract and concentrate
103 informative volatiles from oil samples, to establish correspondences between specific odorants and related
104 sensory attributes, with known metabolic pathways from endogenous or exogenous (from microorganism)
105 enzymes. The results, normalized and interpreted on the basis of target analytes odor thresholds (OT),
106 placed in a more realistic perspective the sensory significance of specific volatiles suggesting an effective
107 strategy of investigation on this subject.
108 However, despite an extensive knowledge on the volatile composition of olive oil, sensory evaluation has
109 not yet been replaced nor supported by any recognized analytical procedure. This is probably due to
110 several aspects, recently discussed by Aparicio *et al.* [29], among which the complexity of sensory
111 perception, the fuzziness of the semantics of some descriptors, and at the same time, the necessity to
112 improve analytical selectivity and resolution along with the pre-concentration procedure, represent
113 objective limits of current methodologies adopted for volatile profiling of olive oil.
114 In this perspective, the present work aims at evaluating whether GC×GC-MS, in combination with both
115 headspace sampling carried out with Solid Phase Microextraction (HS-SPME) and implemented with
116 advanced data elaboration tools, can be a valuable and more informative analytical approach for a high
117 throughput detailed profiling and an effective fingerprinting of the volatile fraction of olive oil in view of its
118 quality assessment.

The chemical blueprint of samples corresponding to regulated defects and authentic EVO samples, provided by IOC, has been adopted as a probe to classify commercial products and to guide an iterative process including sensory evaluation (performed by both an official panel and an internal one), volatiles distribution and their sensory relevance (OT).

2. Materials and Methods

2.1 Reference compounds and solvents

Pure reference compounds for analyte identity confirmation and *n*-alkanes (*n*-C7 to *n*-C30) for Linear Retention Index (I^T_S) determination were from Sigma-Aldrich and Supelco (Milan, Italy). Solvents for *n*-alkanes dilution (toluene and cyclohexane) were HPLC-grade from Riedel-de Haen (Seelze, Germany).

2.2 Samples

The IOC supplied five samples of standard defected oils (*muddy*, *vinegary*, *mold*, *fusty*, and *rancid*); five samples (EVO-1 – EVO-5) were provided by an Official Panel (Stazione Sperimentale per le Industrie degli Oli e dei Grassi, Milano, Italy) after sensory evaluation; 9 samples (EVO-6 – EVO14) were purchased from the local market and evaluated by a trained internal panel (5 people), according to the standardized official method approved by the IOC [14] and reported in Annex XII of ref. [12]. The list of samples analyzed by both research units, along with label descriptions and sensory evaluation is reported in **Table 1**.

2.3 SPME devices and validation

The SPME devices and fibers were from Supelco (Bellefonte, PA, USA). A Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) d_f 50/30 µm, 2 cm length fiber was chosen and conditioned before use as recommended by the manufacturer.

The internal standard (ISTD) loading procedure onto the SPME fiber [30,31] preceded each sampling and was as follows: fiber was inserted into a 20 mL sealed vial containing 500 µL of ISTD (menthol standard solution at 50 ng/mL in dibuthyl phthalate) and exposed to the headspace at 50°C for 40 min. After ISTD loading, the fiber was exposed to the oil sample headspace(2.000 g) at 50°C for 40 min and subsequently introduced into the GC injector for thermal desorption for 10 min at 250 °C. Samples were analyzed in duplicate from the two research units. Precision data referred to intra-laboratory relative standard deviations (RSD %) for retention times of 5% maximum and of 18% on Normalized 2D Peak Volumes (i.e., Cumulative 2D Peak Area normalized over ISTD) for a selection of target analytes (**Table 2**). Inter-laboratory precision was evaluated on Normalized 2D Peak Volumes and was always within a 25% (RSD) range.

2.4. HS-SPME-GC×GC-MS Apolar × Polar set-up

2.4.1 *GC×GC-MS cryogenic modulation optimization*

A loop-type single-jet prototype developed at the University of Udine, was adopted as a cryogenic modulator for the apolar × polar set-up. The heart of the system is a liquid CO_2 valve assembly, equipped with a fixed support to locate easily the loop just at the exit of the CO_2 jet (**Supplementary Figure 1 SF1**), the latter provided by SGE (Australia). The CO_2 jet was periodically activated and deactivated (according to the modulation time set) through the use of compressed air controlled by a 5-way 2 exit solenoid valve (cod. SY3120-5LOU-M5, Clippard, Louvain-La-Neuve, Belgium). The modulation parameters were regulated by a timing controller (H5CX, Omron, Hoofddorp, Netherland).

It is well known that some problems related to the focusing of highly volatile components may occur using CO_2 (instead of liquid nitrogen) as cooling fluid. To circumvent such a limitation, a thick film first dimension column (0.50 µm d_f) was employed to increase the elution temperature of highly volatile compounds. Furthermore, a 1-m capillary segment, with a thin coating (0.10 µm d_f) was used as the modulator loop, to increase the retention capacity during the focusing step. The flow exiting the loop was split between the analytical column and a 1 m × 0.1 mm i.d. of an uncoated capillary segment, thus diverting a part of the first-column effluent to waste (about 50%). Using such a configuration, two main goals were achieved: (*a*) closer to optimal linear velocities generated in both dimensions (about 20 and 115 cm/sec in the ^{1}D and ^{2}D column, respectively) [32], and (*b*) a better reinjection efficiency onto the ^{2}D column, as recently discussed by Tranchida *et al.* [33]. Furthermore, the first modulation stage was performed at the head of the loop (0.25 mm i.d.), while the second was achieved on the 0.1 mm i.d. analytical column (after the split connection), to further reduce the injected bandwidth.

A common orthogonal set, namely apolar × polar, was employed to simplify the peak recognition process, based on the use of Linear Retention Index (I^T_S) values reported in literature and in some commercially available MS databases. A ± 30 range for I^T_S match was applied to confirm or discard less probable mass spectrum similarity match, except for few compounds (10) for which a ± 50 range was considered acceptable, as reported elsewhere for GC×GC application [34]. In fact, the presence of the polar ^{2}D column, in terms of retention, is negligible for the less polar analytes, on the contrary, the more polar components, which have intense interactions, can be strongly retained, causing a significantly shifting in the retention time.

2.4.2. Instrument set up

At the University of Udine, sampling was performed manually following the SPME procedure described in section 2.3 .

The GC×GC system consisted of two GC2010 gas chromatographs on-line coupled to a QP2010 Ultra quadrupole MS (Shimadzu, Kyoto, Japan) operating in EI mode at 70 eV. The transfer line was set to 250°C. The scan range was set to *m/z* 40-350 with a scanning rate of 20,000 amu/s, obtaining a spectra generation frequency of 33 Hz.

The system was equipped with a prototype of loop-type dual-stage thermal modulator, laboratory-made as described in section 2.4.1., cooled with liquid CO_2 with a modulation time of 5 s (4.1 s CO_2 flow on and 0.9 s off) adopted for all experiments. The column set was configured as follows: ^{1}D Rxi-5ms column (similar to 5% phenyl/95% dimethyl polysiloxane)(30 m × 0.25 mm i.d., 0.50 µm d_f) from Restek (Bellefonte, USA) (located in GC oven 1) connected to an Rxi-5ms capillary segment (1.0 m × 0.25 mm i.d. × 0.1 µm d_f,) and then to a Supelcowax-10 (polyethylene glycol) segment (1.2 m × 0.1 mm i.d., 0.10 µm d_f) and to a 1 m × 0.1 mm i.d. uncoated capillary (both columns, provided by Supelco, were located in GC2), by using a fixed outlet capillary column splitter (SGE, Ringwood, Australia). A split ratio of about 1:1 occurred at this point, thus a splitless injection was performed to maintain a satisfactory sensitivity of the entire method [35].

Fibers thermo desorption into the GC injector port occurred using the following set-up: split/splitless in splitless mode held for 1 min, injector temperature 250°C. The carrier gas was helium at a constant flow of 0.7 mL/min (initial head pressure 243 KPa). The temperature program was 40°C (2 min) to 200°C at 3°C/min and to 320°C at 15°C/min. An offset of +10°C was applied in the second oven to reduce wrap-around.

Data were acquired by GCMS Solution (ver. 2.6, Shimadzu, Japan) and processed using GC Chromsquare ver. 1.6 (Shimadzu Europe, Duisburg, Germany). FFNSC 1.6 (Chromaleont, Messina, Italy) and NIST08s MS commercial libraries were used for identification. Statistical analysis was performed with SPSS 14.0 (SPSS Inc. Chicago, Illinois, USA).

For I^T_S determination, the *n*-alkane liquid sample solution was injected using an AOC-20i autosampler (Shimadzu, Japan) under the following conditions: split/splitless injector, split mode, split ratio 1/50, injector temperature 280°C, injection volume 1µL.

2.5 HS-SPME-GC×GC-MS Polar × Apolar set-up

At the University of Torino, volatiles from 2.000 g of oil placed in a 20 mL headspace vial were processed using a HT280T multipurpose sampler (HTA, Brescia, Italy) integrated with an Agilent 6890 GC unit coupled to an Agilent 5975C MS detector (Agilent, Little Falls, DE, USA) operating in EI mode at 70 eV. The GC transfer line was set at 270°C. A *Standard Tune* was used and the scan range was set to m/z 40-240 with a scanning rate of 10,000 amu/s, obtaining a spectra generation frequency of 24 Hz.

The system was equipped with a two-stage KT 2004 loop thermal modulator (Zoex Corporation, Houston, TX) cooled with liquid nitrogen and with the hot jet pulse time set at 250 ms with a modulation time of 5 s adopted for all experiments. Fused silica capillary loop dimensions were 1.0 m length and 0.1 m inner diameter. The column set was configured as follows: ^{1}D SolGel-Wax column (100% polyethylene glycol)(30 m × 0.25 mm i.d., 0.25 µm d_f) from SGE Analytical Science (Ringwood, Austalia) coupled with a ^{2}D OV1701 column (86% polydimethylsiloxane, 7% phenyl, 7% cyanopropyl) (1 m × 0.1 mm i.d., 0.10 µm d_f) from Mega (Legnano, Milan, Italy).

Fibers thermo desorption into the GC injector port occurred using the following set-up: split/splitless in the split mode, split ratio 1:20, injector temperature 250°C. The carrier gas was helium at a constant flow of 0.7 mL/min (initial head pressure 260 KPa). The temperature program was 40°C (1 min) to 180°C at 3°C/min and to 260°C at 20°C/min (5 min).

Data were acquired by Agilent MSD ChemStation ver D.02.00.275 and processed using GC Image GC×GC Software version 2.1b1 (GC Image, LLC Lincoln NE, USA). Statistical analysis was performed with SPSS 14.0 (SPSS Inc. Chicago, Illinois, USA).

For I^T_S determination, the *n*-alkane liquid sample solution was injected using the HT280T sampler (HTA, Brescia, Italy) under the following conditions: split/splitless injector, split mode, split ratio 1/50, injector temperature 280°C, injection volume 1µL.

2.6 Statistical Elaborations and visualization

Principal Component Analysis (PCA), Partial Least Square Discriminant Analysis (PLS-DA) and Orthogonal Partial Least Square Discriminant Analysis (OPLS-DA) were performed with Pirouette software ver. 4.0 (Infometrix, Inc. Bothell, WA, USA). Data were pre-treated by baseline correction through noise subtraction and subsequently pre-processed [36]. Data visualization as heat map was obtained by GENE-E v 3.0.77 (Broad Institute, Inc. Cambridge, MA, USA).

3. Results and Discussion

The analysis strategy started with the untargeted processing of chromatographic data (on raw and pre-processed variables) for sample pre-classification; thus data reduction followed a targeted basis, selecting the most informative analytes whose correlation with sensory defects or technological processing is documented in literature. 2D Patterns obtained with the two-different column combinations adopted for the experiments were successively compared pair-wise to define peculiar distributions of analytes correlated with sensory classification and underline chemical blueprints to be used for a more effective discrimination.

Emphasis will be put on the advantages of the data reduction strategy based on both: (*a*) structured and detailed 2D patterns provided by GC×GC-MS and (*b*) higher informative potential of potent odorants within the group of volatiles.

3.2 Untargeted fingerprinting

To fully exploit the informative content of the three dimensional GC×GC-MS data set (^{1}D and ^{2}D retention times and MS fragmentation patterns) the so-called *peak features* methods, have demonstrated

to be effective and, although to a different extent, sensitive enough in monitoring variations in the chemical pattern across complex samples [10].

Among the existing methods, *Comprehensive Template Matching* fingerprinting (CTMF) [34] offers the possibility to fully exploit the data matrix generated by GC×GC-MS, by extending correspondences not only to ^{1}D and ^{2}D retention, but also to the similarities with the MS fragmentation pattern adopting the NIST MS Search algorithm [37].

In the present study, 2D patterns from oil samples analyzed with the two column combinations (apolar × polar and polar × apolar) in different laboratories, for a total of 76 2D plots (19 sample x 2 replicates x 2 laboratories), were processed by CTMF and reliable *peak features* collected in a cumulative template used to align 2D peaks across sample chromatograms and extract mass quantitative descriptors (Normalized 2D Peak Volumes) for the comparative analysis.

The polar × apolar column set provided 452 *peak features,* while the apolar × polar set gave 395. The list of *peak features* and related information (^{1}D and ^{2}D retention times, I^{T}_{S} in the ^{1}D, Normalized 2D Peak Volumes and MS fragmentation pattern) are provided as supplementary data (**Supplementary Table ST1**).

The resulting Normalized 2D Peak Volumes, referred to the 452 and 395 untargeted analytes, are visualized as a heat map in **Supplementary Figure 2** (**SF2a** apolar × polar and **SF2b** polar × apolar). Variables for the heat map construction (i.e., 2D Peak Volumes) were pre-processed by subtracting row mean and dividing by standard deviation.

The two raw dataset (corresponding to the apolar × polar and polar × apolar) were submitted to Principal Component Analysis (PCA) to see whether samples would be successfully clustered according to their sensory quality (*lampante*, VO and EVO).

As shown in **Figure 1**, considering raw data (without scaling), for both column configurations the variance described by the first three components is respectively of 63% (**Figure 1a** apolar × polar) and 86% (**Figure 1b** polar × apolar). However, the model based on the untargeted approach does not discriminate samples according to their commercial classification: i.e., *lampante* oils (*muddy*, *mold*, *vinegary* and *fusty*) are not well separated from commercial EVOs (EVO-1 to 14). When the PCA is performed on pre-treated variables (baseline correction and auto-scaling) the total variance approaches the 72 % (**Figure 1c** and **1d**) but, again, the related models do not improve discrimination attitudes. This is probably due to a high noise level from variables that are not representative in the description of the sensory quality of samples under study.

The successive steps followed a different strategy: untargeted *peak features* were identified on the basis of MS fragmentation patterns, I^{T}_{S} in the ^{1}D and, where possible, authentic standard confirmation. The list of 261 target analytes is provided as **Supplementary Table 2**, along with their odor descriptors, known correlation with sensory defects or quality (Mo for *mold*; M for *muddy*; F for *fusty*; V for *vinegary*; R for *rancid*; Fr for *fruity*) [27] and the MS matching %.

The resulting 2D patterns were submitted to sequential pair-wise comparisons to reveal peculiar quali-quantitative distributions of informative chemicals to be correlated with the sensory quality of samples.

3.2 Potentials of 2D pattern recognition in revealing the chemical blueprint of olive oil

Volatiles that contribute to the whole aroma of virgin olive oil belong to several classes with a prevalence of polar functionalities: aldehydes, ketones, alcohols, esters, hydrocarbons, furans, lactones, and their relative distribution depends on several, concurrent, parameters. The resulting chemical pattern is quite complex and the identification of an unequivocal sample fingerprint for sensory quality assessment is challenging.

Cultivar, geographical origin, fruit ripeness, processing practices, and storage significantly modify the distribution of volatiles [39], as well as enzymatic activity, such as that of the lipoxygenase pathway (LOX) [40]. In addition, the technological process, and in particular the crushing and malaxation steps, may influence volatiles biosynthesis by promoting/inhibiting different pathways [24,25,41,42].

Sensory-relevant and diagnostic analytes may be hidden, or their reliable identification limited by method LOAV (Limit of Odour Activity Value) [4], a useful parameter recently introduced in sensomics, given by the OT divided by analyte LOQ. By definition, LOAV values above 1 refer to sensitive methods capable of efficient and quantitative assessment of odorants, above their odor threshold, while LOAV below 1 indicates the concentration limit down to which an odorant can be identified but not quantified.

In this context, GC×GC undoubtedly is a strategic choice, able to provide highly detailed, sensitive and structured 2D pattern that acts as a peculiar sample signature for chemical fingerprinting [9,43].

In this perspective, the visual comparison tool of both elaboration software adopted in this study, was used as a support for the 2D pattern recognition and peak finding; each reference defected standard oil from IOC (Mo, M, F, V and R) was pair-wise compared to authentic EVO samples certified by the panel (EVO-1, EVO-5, EVO-8, EVO-10, EVO-11 and EVO-14) to localize relevant discriminating *peak features* across patterns. **Figure 2** reports a schematic summary of the sequential process adopted together with two GC×GC patterns of volatiles. Analytes correlated with specific defects were thus collected in a fingerprint template that was successively applied to the 2D pattern of slightly defected oils (EVO-2, EVO-3, EVO-4, EVO-6, EVO-7, EVO-9, EVO-12 and EVO-13). This last step was implemented to validate the effectiveness of selected analytes/probes, and to see whether or not the prediction of a certain combination of defects was possible on the basis of the chemical fingerprint.

The results show good consistency within the column set in terms of quantitative descriptors (see also section *2.3*). Target analytes relative abundance (normalization was done on menthol as ISTD) did not exceed the ± 25% of RSD, allowing the cross-validation of the comparative analysis, although, several diagnostic analytes were not detected by the polar × apolar set; as in the case of ethyl propanoate (25),

methyl butanoate (28), (3Z)-hex-3-enal (45), 6-methylhept-5-en-2-ol (137), o-guaiacol (178) and (2Z)-non-2-enal (217).

Looking at fingerprint analytes related to defected and/or to EVO samples (**Supplementary Table 2** - Sensory defect column header), the pattern recognition strategy proved to be highly reliable: known marker compounds already reported in literature [21-27,29] were all positively matched and several others, with a discriminating potential and sensory contribution (very low OTs) could be added.

Some alcohols, like butan-1-ol (12), pentan-3-ol (23) and (3Z)-hex-3-en-1-ol (66) were found to be also present in defected samples (e.g *vinegary*); carbonyls like pentan-2-one (16), 2-methylbutanal (20), (3E)-octa-3,5-dien-2-one (185), (3E)-6-methylhepta-3,5-dien-2-one (191) were included, with the last two targets with very low OTs (0.0005 and 0.38 ng/g respectively), and two esters: ethyl octanoate (230), ethyl decanoate (256). To the *fusty* fingerprint, (3Z)-hex-3-en-1-ol (66) and propyl acetate (26) were also added while for *mold*, (2E)-2-methylbut-2-enal (40), (3E)-octa-3,5-dien-2-one (185) and (3E)-6-methylhepta-3,5-dien-2-one (191) were discriminant.

Positive matches and relative Normalized 2D Peak Volumes collected for all the data set and reported in **Table 2** were submitted to a Partial Least Square Discriminant Analysis (PLS-DA) to prove the effectiveness of the blueprint analytes selected (for whom experimental OT values in lipophilic media were available in literature [16, 27]) in predicting the presence of certain quality attributes in a sample,.

This algorithm is a PLS regression where the response variable is categorical and indicates samples classes/categories. Supervised pattern recognition techniques are effective to build a model to be used for future classifications when classification is known "*a priori*". PLS-DA enables both classification and discrimination, and compared to other algorithms (SIMCA or KNN) its power rely on the ability to explain differences between overall class properties and related most informative class analytes.

The results are visualized in **Figure 3**, and refer a good separation between defected (arbitrarily categorized as *class 1*) grouped on the right side of the graph and authentic EVO samples (*class 2*) on the left side. In fact, according to the IOC classification codes of regulated sensory defects (M, V, Mo and F), PLS-DA results show that the first three PCs (**Figure 3a**) afford to effectively classify the two classes with the 56.17% of explained variance, although the chemical pattern of the IOC defected samples differs significantly from defected EVOs. **Figure 3b** (loading plot) shows the variables that are more correlated to the defects and *vice versa*.

Interestingly, analytes that prevail are those strongly correlated with oxidation and enzymatic activity (mono and poly-unsaturated aldehydes, esters formed as secondary products of fermentation and some 2 methyl-ketones).

The Orthogonal Signal Correction (OPLS) on PLS-DA results enables to isolate within the data matrix, the most relevant information according to intra and inter-class samples classification.

OPLS-DA results (not shown) indicated that IOC defected samples were highly correlated with hexanal (46) (Mo and V), octane (53) (V), 6-methylhept-5-en-2-one (126) (V, Mo and F), nonanal (196) (M), while for the fusty (F) reference sample and EVOs (3-5-8-10-11-14) other variables referred a better correlation (i.e., 2E,4Z-hept-2,4-dienal (125), 2E-hept-2-enal (110), octan-2-one (134), pentan-3-ol (23), 3z-hexe-3-1-ol (66) and octanal (138)).

The model was internally full cross-validated (leave-one-out method) and calculated sensitivity and specificity were of 100%. The model sensitivity refers to the percentage of acceptance related to its own objects while specificity refers to the percentage of the objects of another class rejected by the model. Such an over fitting was therefore expected because of the statistically limited number of samples considered; this limitation would be overcame by appropriate sampling extending the number of informative samples per each class.

However, although the combination of HS-SPME and GC×GC-MS allowed to improve method LOD and LOQs, compared to conventional 1DGC-MS approaches, resulting in an extended pattern of informative volatiles, a further step was necessary to develop a predictive method for quality assessment. In this perspective, the principles of sensomics were implemented to see whether or not it may be successful in this context.

3.3 Improving the informative potential of the chemical blueprint: toward sensomics

The sensory quality of each sample not only depends on the presence or absence of specific compounds, but it is related to their odour potency and the resulting concentration in the final product. In this perspective, OT can be used to rank analytes as a function of the sensory potency, although with some limits due to the non-linear dose-response relationship between the perception and the odorant concentration in the sample as demonstrated, and critically discussed, by several authors [45-47]..

Therefore, keeping in mind that the pattern of volatiles obtained by HS-SPME sampling reflects, not only analytes concentration, but also their volatility and/or affinity for fiber's coating; 2D Peak Volumes were normalized by OT values from literature. This approach that impacts on the magnitude of variables according with their odour potency, emphasizes the role played in the classification by components with very low OT values or, on the other hand, those with a higher response, reasonably more abundant in the sample.

PLS-DA performed on the resulting data matrix (**Figure 4a** and **4b**) shows a better classification of samples; the first three PCs explain 71.28% of the total and the most informative analytes (**Figure 4b**) for authentic EVOs resulted in: pent-1-en-3-one (14), pent-1-en-3-ol (15), (2E)-hex-2-enal (63) while for defected samples were: butan-1-ol (12), 3-methylbutan-1-ol (31), hexanal (46), octane (53), heptanal (85), (2E)-hept-2-enal (110), oct-1-en-3-ol (131), nonanal (196), (2E)-non-2-enal (218), (2E)-dec-2-enal (245).

It is worthy of note that this classification model is mostly driven by markers of defects rather than quality indicators, due to the contribution of IOC reference samples to the defected class. These samples are in fact characterized by a peculiar chemical pattern. Therefore, it can be stated that, although the study of reference defected samples is important to understand any phenomenon, a more "comprehensive" sampling (including slightly defected oils and real-world samples) gives more realistic pictures facilitating the identification of relevant analytes responsible for a particular fingerprint.

4. Conclusions and future perspectives

The present study, although not conclusive, emphasizes the analytical advantages of GC×GC-MS in terms of sensitivity, reproducibility (evaluated by cross-validation between laboratories and instrumental set-up) and information potential of the 2D patterns produced, proposing a productive investigation strategy for a reliable quality assessment of EVO and VO oils.

Reliable results were obtained on a larger pattern of chemicals in the perspective of samples classification based on sensory quality by combining the effectiveness of the analytical procedure (including volatile extraction by HS-SPME) to provide a large and reliable data set of potentially discriminating analytes and adopting sensomics data interpretation principles (mostly based on sensory active compounds and their related odour potency).

The results were validated from two different points of view: (*a*) by cross-comparison between laboratories working with different instrumentation and software packages and (b) by confirming analytes informative potential with existing studies and available literature. The role played by those chemicals specifically revealed by the presented approach will be the focus of future research aimed at their quantification to obtain reference blueprints of defects. In this perspective the adoption of alternative HCC-HS sampling techniques, connoted by a higher concentration capacity (such as Headspace Sorptive Extraction – HSSE with different polar and/or apolar extraction polymers), which improve fingerprinting sensitivity and enable true quantitation (as for example by Multiple Headspace Extraction – MHE) of key-odorants [43] would be of interest in the perspective of an objective evaluation of EVO oils quality.

This approach, applied to a wider and strictly structured experimental design (considering more variables, such as cultivar, geographical origin, different defect intensity etc), supported by more than one official Panel, can allow to robustly and reliably characterize specific markers and related characteristics concentration windows, and thus be used as a valuable tool to support, or even replace, sensory evaluation.

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Supplementary data

Supplementary Figure 1. Loop-type single-jet prototype developed at the University of Udine. For details see text.

Supplementary Figure 2. Heat map based on untargeted *peak features* detected by the apolar × polar (**ST1a**) and the polar × apolar (**ST1b**) column set. Absolute 2D Peak Volumes are normalized across each sample set by subtracting row mean and dividing by standard deviation. *Peak features* along columns follow the retention time ordering.

Supplementary Table 1 List of *peak features* and related information (^{1}D and ^{2}D retention times, I^T_S in the ^{1}D, Normalized 2D Peak Volumes and MS fragmentation pattern).

Supplementary Table 2 List of identified compounds in the apolar × polar set, along with ^{1}D and ^{2}D retention times, experimental and library reported I^T_S, known correlation with sensory defects, odor descriptors and MS similarity matching (%). Mo: *mold*; M: *muddy*; F: *fusty*; V: *vinegary*; R: *rancid*; Fr: *fruity.* Defects marked with * refer to correlation highlighted using the proposed sequential process.

Figure captions

Figure 1. PCA score plots performed on untargeted *peak features* resulted from GC×GC-MS carried out on the apolar x polar (**1a** and **1c**) and polar x apolar column set (**1b** and **1d**). Raw data visualization **1a)** explained variance 63%,**1b)** explained variance 86.2%. PCA on pre-treated (baseline correction) and auto-scaled data **1c)** explained variance 71.95% and **1d)** explained variance 72.56%. For data interpretations see text.

Figure 2. Scheme of the sequential process applied in this study.

Figure 3. Internally cross-validated PLS-DA model and resulting score (**3a**) and loadings (**3b**) plots performed on the Normalized 2D peak volumes of the compounds listed in **Table 2**. Explained variance on the first three PCs is 56.17%; baseline correction and pareto pre-processing were performed.

Figure 4. Internally cross-validated PLS-DA model and resulting score (**4a**) and loadings (**4b**) plots performed on the Normalized 2D peak volumes divided by the OTs on compounds listed in **Table 2**. Explained variance on the first three PCs is 71.28%, baseline correction and pareto pre-processing were performed.

Table captions

Table 1. Description of the olive oil samples.

Table 2. List of compounds adopted in the chemical blueprint of samples. ^{1}D and ^{2}D retention times, experimental I^T_S, known correlation with sensory defect, odor threshold values (OT in mg/kg) and normalized peak volume (multiplied per 10^5) are reported for both apolar × polar and polar × apolar (in *italic*) column set.

Table 1. Description of the olive oil samples.

Sample Name	Type	Sensory Analysis		Additional label information
		Panel	Descriptors	
Musty	Lampante oil	Official	Intense Musty	IOC standard
Vinegary	Lampante oil	Official	Intense Vinegary	IOC standard
Fusty	Lampante oil	Official	Intense Fusty	IOC standard
Mould	Lampante oil	Official	Intense Mould	IOC standard
Rancid	Lampante oil	Official	Moderate Rancid	IOC standard
EVO-1	Commercial EVO (small production)	Official	EVO	Declared to be produced with Italian olives
EVO-2	Commercial EVO (small production)	Official	Mould and rancid	Declared to be produced with Italian olives
EVO-3	Commercial EVO (small production)	Official	Fusty and muddy	Declared to be produced with Spanish olives
EVO-4	Commercial EVO (small production)	Official	Fusty, muddy, vinegary	Declared to be produced with Spanish olives
EVO-5	Commercial EVO (small production)	Official	EVO	Declared to be produced with Greece olives
EVO-6	Commercial EVO (industrial production)	Internal	Fusty/muddy	Declared to be produced with EU olives
EVO-7	Commercial EVO (industrial production)	Internal	Fusty/muddy	Declared to be produced with EU olives
EVO-8	Commercial EVO (industrial production)	Internal	EVO	Declared to be produced with EU olives
EVO-9	Commercial EVO (industrial production)	Internal	Slightly fusty	Declared to be produced with Italian olives
EVO-10	Commercial EVO (industrial production)	Internal	EVO	Declared to be produced with Italian olives
EVO-11	Commercial EVO (industrial production)	Internal	EVO	Declared to be produced with EU olives
EVO-12	Commercial EVO (industrial production)	Internal	Intense rancid	Declared to be produced with Italian olives
EVO-13	Commercial EVO (industrial production)	Internal	EVO/slightly rancid	Declared to be produced with Italian olives
EVO-14	Commercial EVO (small production)	Internal	EVO	Declared to be produced with Slovenian olives

Table 2. List of compounds adopted in the chemical blueprint of samples. ^{1}D and ^{2}D retention times, experimental I^{T}_{S}, known correlation with sensory defect, odor threshold values (OT in mg/kg) and normalized peak volume (multiplied per 10^{5}) are reported for both apolar × polar and polar × apolar (in *italic*) column set.

ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory defect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
1	ethanol	2.78	1.72	552	V	30	3.7	1.0	2.8	4.3	6.3	2.1	6.2	0.0	114.9	2.6	79.0	3.4	29.2	24.1	18.5	12.3	29.7	8.5	5.5
		7.67	*1.14*	*883*			*4.7*	*1.3*	*3.3*	*4.0*	*6.3*	*2.8*	*5.6*	*0.0*	*65.0*	*4.0*	*75.7*	*3.1*	*37.0*	*14.9*	*18.0*	*10.0*	*20.0*	*8.8*	*6.2*
6	butanal	6.85	1.05	607	F/M	0.018	11.3	0.0	0.3	2.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		7.00	*2.48*	*857*			*14.2*	*0.0*	*0.4*	*3.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
7	ethyl acetate	7.52	0.96	613	F/V	0.94	18.9	17.2	5.3	4.7	0.0	0.0	0.0	9.4	16.9	0.0	85.7	40.2	12.4	10.7	8.5	2.8	0.0	3.2	0.0
		6.75	*1.35*	*850*			*23.8*	*18.9*	*6.2*	*4.3*	*0.0*	*0.0*	*0.0*	*9.2*	*9.6*	*0.0*	*82.1*	*37.1*	*15.8*	*6.6*	*8.3*	*2.3*	*0.0*	*3.2*	*0.0*
9	acetic acid	8.11	1.66	622	F/V/R	0.5	12.5	23.7	18.4	32.9	12.6	4.6	12.6	21.0	194.2	5.6	3.6	37.0	13.5	5.0	2.9	6.7	0.7	1.1	8.1
		28.50	*0.97*	*1457*			*15.7*	*26.0*	*21.5*	*30.7*	*12.6*	*6.2*	*11.2*	*20.8*	*109.9*	*8.6*	*3.4*	*34.1*	*17.1*	*5.0*	*2.9*	*7.3*	*0.7*	*1.0*	*6.8*
12	butan-1-ol	9.47	2.92	648	V/M *	0.15	1.6	4.0	0.0	17.1	0.0	0.3	1.3	1.8	0.6	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.2	0.0
		14.42	*1.26*	*1120*			*1.5*	*3.6*	*0.0*	*16.0*	*0.0*	*0.4*	*1.1*	*1.8*	*0.4*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.2*	*0.0*
13	3-methylbutanal	9.52	0.96	649	F/V	0.013	0.0	1.2	0.0	0.1	0.0	0.0	0.0	0.6	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		8.75	*2.61*	*927*			*0.0*	*1.5*	*0.0*	*0.1*	*0.0*	*0.0*	*0.0*	*0.6*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
14	pent-1-en-3-one	10.61	1.39	668	M	0.00073	0.7	1.6	0.6	2.4	1.4	29.5	2.0	6.1	0.4	4.6	0.9	24.3	1.5	0.0	3.4	2.8	0.3	0.5	8.9
		10.17	*1.47*	*983*			*0.9*	*1.8*	*0.8*	*2.2*	*1.4*	*39.6*	*1.8*	*6.0*	*0.2*	*7.1*	*0.9*	*22.4*	*1.9*	*0.0*	*3.3*	*2.3*	*0.2*	*0.6*	*10.1*
15	pent-1-en-3-ol	10.71	2.77	670	F/Mo	0.4	1.6	6.8	1.2	11.3	11.6	18.0	8.4	7.5	9.6	15.1	6.0	8.3	9.4	11.7	11.8	10.8	7.4	5.3	4.5
		15.08	*1.30*	*1135*			*2.0*	*7.5*	*1.4*	*10.6*	*11.6*	*24.2*	*7.5*	*7.4*	*8.2*	*20.2*	*5.8*	*7.6*	*11.9*	*7.3*	*11.5*	*8.8*	*6.1*	*5.4*	*5.1*
16	pentan-2-one	10.77	1.18	671	V	70	0.5	1.8	0.5	0.9	0.0	0.0	0.5	1.0	0.0	1.8	0.5	0.0	12.3	23.8	0.0	0.0	0.4	1.9	0.8
		8.84	*1.47*	*930*			*0.7*	*1.9*	*0.6*	*0.8*	*0.0*	*0.0*	*0.4*	*0.9*	*0.0*	*2.4*	*0.5*	*0.0*	*15.6*	*14.8*	*0.0*	*0.0*	*0.3*	*1.9*	*0.9*
20	2-methylbutanal	11.02	1.15	676	V/M *	0.01	3.8	16.0	7.5	9.3	0.7	16.7	20.5	18.2	5.1	0.0	16.3	0.0	0.0	0.0	21.1	18.9	8.4	19.2	0.0
		4.34	*1.09*	*750*			*4.7*	*17.6*	*8.7*	*8.7*	*0.7*	*22.4*	*18.3*	*18.0*	*4.4*	*0.0*	*15.6*	*0.0*	*0.0*	*0.0*	*20.5*	*15.3*	*6.9*	*19.8*	*0.0*
23	pentan-3-ol	11.75	4.97	689	V	9	0.5	14.0	0.0	0.0	0.0	0.8	0.0	0.0	1.2	0.0	1.8	0.8	0.8	1.0	0.0	0.0	0.0	0.0	0.0
		13.00	*1.35*	*1078*			*0.6*	*11.0*	*0.0*	*0.0*	*0.0*	*1.1*	*0.0*	*0.0*	*1.0*	*0.0*	*1.7*	*0.7*	*1.0*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
25	ethyl propanoate	12.10	0.93	696	F	0.1	4.8	0.4	0.4	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
28	methyl butanoate	12.43	1.05	702	F	0.06	0.8	0.0	0.0	4.4	0.0	0.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
31	3-methylbutan-1-ol	13.46	2.62	720	F/M/Mo	0.1	2.4	8.7	1.0	2.9	0.0	1.0	5.3	18.1	23.1	3.1	15.5	11.5	4.9	8.0	3.3	4.1	2.8	3.6	1.2
		18.35	*0.94*	*1215*			*3.0*	*9.6*	*1.1*	*2.7*	*0.0*	*1.4*	*4.7*	*17.9*	*19.8*	*4.1*	*14.9*	*10.6*	*6.2*	*4.9*	*3.2*	*3.3*	*2.3*	*3.7*	*1.4*
32	(2E)-pent-2-enal	13.53	1.93	721	V	0.3	5.9	12.9	6.9	21.4	10.5	4.4	3.4	5.7	1.2	4.5	1.6	0.0	1.2	0.5	1.4	1.1	2.9	1.3	1.0
		14.08	*1.52*	*1110*			*7.4*	*14.2*	*8.1*	*20.0*	*10.5*	*6.0*	*3.0*	*5.6*	*1.0*	*6.0*	*1.5*	*0.0*	*1.5*	*0.3*	*1.4*	*0.9*	*2.4*	*1.3*	*1.1*
39	pentan-1-ol	14.88	2.92	746	F/M/V	3	1.0	2.1	0.9	4.5	1.9	1.0	3.5	2.1	2.0	3.7	4.0	5.8	1.0	6.0	1.3	1.4	3.4	7.4	0.0
		19.00	*1.35*	*1231*			*1.0*	*2.1*	*0.9*	*4.5*	*1.9*	*1.0*	*3.5*	*2.1*	*2.0*	*3.7*	*4.0*	*5.8*	*1.0*	*6.0*	*1.3*	*1.4*	*3.4*	*7.4*	*0.0*
45	(3Z)-hex-3-enal	15.86	1.66	764	Fr	0.003	0.0	3.2	1.3	4.7	11.8	14.2	7.6	2.7	0.0	5.3	1.5	1.7	3.4	0.7	1.8	3.9	28.9	15.8	9.4
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
46	Hexanal	15.94	1.33	773	F/Mo/V/R	0.08	92.8	170.7	209.1	7.6	59.8	20.6	37.1	44.7	14.7	22.2	15.8	16.3	25.1	32.9	29.4	33.4	212.8	37.0	13.0
		12.25	*1.77*	*1054*			*116.7*	*187.7*	*244.7*	*7.1*	*59.8*	*27.7*	*33.1*	*44.2*	*12.6*	*29.6*	*15.2*	*15.0*	*31.9*	*20.4*	*28.6*	*27.1*	*175.8*	*38.0*	*14.8*
48	ethyl butanoate	17.10	0.93	786	F	0.03	5.2	0.7	0.0	31.3	0.0	0.0	0.0	1.6	1.6	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.0
		10.59	*1.77*	*1000*			*6.6*	*0.8*	*0.0*	*29.2*	*0.0*	*0.0*	*0.0*	*1.6*	*1.4*	*0.0*	*0.0*	*0.0*	*0.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*

Table 3. continued.

ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory defect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
49	oct-1-ene	17.18	0.54	787	M	0.08	3.9	1.6	2.7	1.5	0.0	0.0	3.9	4.8	7.9	0.7	0.0	0.0	0.0	0.0	0.0	0.0	8.4	0.0	0.0
		6.09	*1.68*	*820*			*4.9*	*1.8*	*3.2*	*1.4*	*0.0*	*0.0*	*3.5*	*4.7*	*6.7*	*0.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*6.9*	*0.0*	*0.0*
52	butyl acetate	17.60	1.05	795	F	0.3	0.0	0.0	0.0	9.3	0.0	0.0	0.3	1.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		12.00	*1.73*	*1046*			*0.0*	*0.0*	*0.0*	*8.7*	*0.0*	*0.0*	*0.2*	*1.1*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
53	octane	17.68	0.51	800	F/V/R	0.94	51.9	115.2	65.3	75.4	0.4	0.9	21.9	80.2	51.1	9.3	24.1	31.8	8.6	7.2	7.5	12.2	20.4	42.7	0.0
		5.59	*1.89*	*800*			*65.3*	*126.7*	*76.4*	*70.3*	*0.4*	*1.2*	*19.5*	*79.2*	*43.8*	*12.4*	*23.1*	*29.3*	*10.9*	*4.5*	*7.3*	*9.9*	*16.9*	*43.9*	*0.0*
63	(2E)-hex-2-enal	19.11	1.84	822	Mo/V/F/R	0.42	1.0	1.7	1.9	8.2	565.4	582.0	396.0	37.0	10.4	209.1	218.4	209.0	545.4	444.5	565.9	425.7	406.8	391.3	782.0
		18.00	*1.64*	*1208*			*1.2*	*1.8*	*2.3*	*7.7*	*565.4*	*781.4*	*353.0*	*36.5*	*8.9*	*279.5*	*209.2*	*192.6*	*692.1*	*275.2*	*551.0*	*345.5*	*336.1*	*402.5*	*886.9*
64	(3E)-hex-3-en-1-ol	19.31	3.69	826	F/R/V	6	0.7	0.4	0.6	0.8	1.6	0.6	0.7	1.4	2.6	2.6	3.7	2.6	1.5	2.1	1.3	3.0	1.9	5.9	2.2
		24.08	*1.30*	*1350*			*0.9*	*0.5*	*0.6*	*0.7*	*1.6*	*0.8*	*0.6*	*1.3*	*2.2*	*3.5*	*3.6*	*2.4*	*2.0*	*1.3*	*1.3*	*2.4*	*1.6*	*6.1*	*2.5*
66	(3Z)-hex-3-en-1-ol	19.55	3.25	830	V/F	1.5	1.4	0.6	0.0	15.8	20.0	9.3	1.2	73.1	75.8	58.2	71.8	83.2	34.5	52.1	49.8	42.7	5.1	6.8	18.4
		24.92	*1.35*	*1369*			*1.8*	*0.6*	*0.0*	*14.7*	*20.0*	*12.5*	*1.1*	*72.1*	*64.9*	*77.8*	*68.8*	*76.7*	*43.8*	*32.2*	*48.5*	*34.6*	*4.2*	*7.0*	*20.8*
68	(2E)-hex-2-en-1-ol	19.89	3.58	837	V	5	0.9	3.9	0.0	0.0	49.0	55.8	115.9	9.8	26.1	80.3	61.0	87.9	54.9	81.7	33.4	65.3	14.8	194.5	19.1
		25.92	*1.26*	*1392*			*1.1*	*4.3*	*0.0*	*0.0*	*49.0*	*75.0*	*103.3*	*9.7*	*22.4*	*107.3*	*58.5*	*81.0*	*69.6*	*50.6*	*32.6*	*53.0*	*12.2*	*200.0*	*21.6*
74	hexan-1-ol	20.63	2.71	850	Fr	0.4	1.3	4.6	1.3	19.2	0.0	25.1	33.6	23.8	40.3	34.9	59.4	98.5	54.0	82.0	39.3	54.1	11.0	79.5	17.5
		18.17	*1.81*	*1212*			*1.7*	*5.0*	*1.5*	*17.9*	*0.0*	*33.7*	*30.0*	*23.5*	*34.6*	*46.6*	*56.9*	*90.8*	*68.6*	*50.8*	*38.2*	*43.9*	*9.0*	*81.8*	*19.8*
80	heptan-2-one	21.85	1.27	872	V	0.3	3.8	10.0	7.6	7.5	0.5	0.3	0.7	2.5	0.9	3.4	2.7	1.6	0.6	0.6	0.0	1.4	2.1	0.0	0.0
		16.42	*1.89*	*1169*			*4.8*	*10.9*	*8.9*	*7.0*	*0.5*	*0.4*	*0.6*	*2.5*	*0.8*	*4.5*	*2.6*	*1.5*	*0.8*	*0.4*	*0.0*	*1.1*	*1.7*	*0.0*	*0.0*
85	heptanal	22.35	1.24	881	R	0.5	10.8	6.9	16.7	7.3	1.6	0.3	3.5	5.6	1.6	1.1	2.5	0.0	0.0	0.0	0.0	0.9	8.0	2.0	0.0
		16.50	*1.89*	*1161*			*13.6*	*7.6*	*19.5*	*6.8*	*1.6*	*0.4*	*3.1*	*5.5*	*1.4*	*1.5*	*2.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*6.6*	*2.0*	*0.0*
86	ethyl pentanoate	22.68	0.96	887	M	0.0015	1.3	0.9	0.0	0.0	0.0	0.0	0.0	0.0	0.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.8	1.3	0.0
		23.08	*2.27*	*1327*			*1.7*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*1.3*	*0.0*
103	(2Z)-hept-2-enal	24.28	1.72	917	R	0.042	0.7	0.8	1.2	2.4	0.5	0.0	0.2	0.0	0.0	0.6	0.0	0.0	0.0	0.0	0.0	2.1	1.3	3.1	0.0
		22.58	*1.77*	*1315*			*0.8*	*0.9*	*1.4*	*2.2*	*0.5*	*0.0*	*0.2*	*0.0*	*0.0*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.7*	*1.0*	*3.1*	*0.0*
110	(2E)-hept-2-enal	24.78	1.87	926	Mo/R	0.005	14.8	14.0	39.6	2.8	12.0	2.3	9.9	12.3	4.6	4.8	3.6	3.8	0.9	3.5	2.4	0.0	21.1	0.0	0.4
		22.67	*1.81*	*1317*			*18.6*	*15.5*	*46.3*	*2.6*	*12.0*	*3.1*	*8.8*	*12.2*	*4.0*	*6.4*	*3.5*	*3.5*	*1.2*	*2.2*	*2.3*	*0.0*	*17.4*	*0.0*	*0.5*
122	(2Z,4E)-hepta-2,4-dienal	26.30	2.98	955	R/Mo/F	0.36	0.7	1.7	0.6	0.7	1.2	0.0	2.7	11.6	0.0	0.0	4.1	0.0	0.0	0.0	0.0	0.0	0.0	1.7	0.0
		28.58	*1.60*	*1455*			*0.9*	*1.9*	*0.8*	*0.7*	*1.5*	*0.0*	*2.4*	*11.5*	*0.0*	*0.0*	*4.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.8*	*0.0*
123	oct-1-en-3-one	26.44	1.42	958	Mo	0.01	13.5	11.0	37.8	27.1	9.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	14.4	0.0	0.0	0.0	0.0	0.0
		21.58	*1.89*	*1292*			*16.9*	*12.1*	*44.3*	*25.3*	*9.1*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*8.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
125	(2E,4Z)-hepta-2,4-dienal	26.63	2.77	961	R/Mo/F	10	6.6	15.7	8.3	24.4	25.7	4.2	0.3	0.6	2.5	4.0	2.6	4.3	1.0	4.3	4.3	2.9	5.5	0.0	0.0
		28.66	*3.24*	*1457*			*8.3*	*17.2*	*9.7*	*22.8*	*25.7*	*5.7*	*0.3*	*0.6*	*2.2*	*5.3*	*2.5*	*4.0*	*1.3*	*2.6*	*4.2*	*2.4*	*4.6*	*0.0*	*0.0*
126	6-methylhept-5-en-2-one	26.69	1.63	962	Mo/F/R	1	148.2	88.6	88.6	70.7	2.1	0.8	4.7	11.0	4.4	4.0	2.9	3.0	0.0	0.0	0.0	1.9	4.5	2.0	0.0
		23.17	*1.85*	*1329*			*186.3*	*97.5*	*103.7*	*66.0*	*2.1*	*1.1*	*4.2*	*10.8*	*3.7*	*5.3*	*2.8*	*2.8*	*0.0*	*0.0*	*0.0*	*1.5*	*3.7*	*2.1*	*0.0*
131	oct-1-en-3-ol	27.04	2.26	969	Mo	0.05	4.0	3.1	11.2	26.2	2.6	0.5	2.1	2.0	1.2	1.3	1.6	0.0	0.5	0.0	0.0	0.0	1.2	2.0	0.0
		27.83	*1.47*	*1437*			*0.6*	*0.3*	*1.0*	*1.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
134	octan-2-one	27.35	1.27	975	V	0.51	4.4	4.9	7.3	5.9	0.5	0.4	0.6	1.0	1.6	3.6	2.6	0.9	0.0	0.9	1.7	0.8	1.3	0.5	0.0
		20.83	*2.06*	*1274*			*0.0*	*0.0*	*0.1*	*0.2*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*

Table 3. continued.

ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory défect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
135	(2E,4E)-hepta-2,4-dienal	27.38	2.98	976	R/Mo/F	0.36	3.3	6.7	3.1	12.8	16.3	1.0	1.0	3.6	0.8	0.0	0.0	2.1	0.0	1.4	2.6	1.6	3.4	1.6	0.0
		28.75	*1.73*	*1459*			*4.1*	*7.3*	*3.6*	*11.9*	*16.3*	*1.3*	*0.9*	*3.6*	*0.7*	*0.0*	*0.0*	*1.9*	*0.0*	*0.8*	*2.6*	*1.3*	*2.8*	*1.6*	*0.0*
137	6-methylhept-5-en-2-ol	27.70	2.23	982	Mo	2	1.0	0.6	1.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
138	octanal	27.85	1.27	984	Mo/R	0.32	5.2	8.0	13.1	12.9	1.2	0.3	0.0	79.6	1.1	58.8	54.1	60.6	48.1	80.9	10.7	63.9	4.8	2.7	0.0
		21.08	*2.02*	*1280*			*6.5*	*8.8*	*15.3*	*12.0*	*1.2*	*0.3*	*0.0*	*78.6*	*0.9*	*78.6*	*51.8*	*55.9*	*61.1*	*50.1*	*10.4*	*51.8*	*3.9*	*2.8*	*0.0*
140	octan-2-ol	27.94	1.63	986	Mo	0.1	0.7	0.0	3.5	1.3	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		25.50	*1.60*	*1383*			*0.8*	*0.0*	*4.1*	*1.2*	*0.0*	*0.0*	*0.0*	*0.0*	*0.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
149	hexyl acetate	28.52	1.08	997	Fr	1.04	5.4	5.2	4.4	1.1	2.0	0.8	5.5	20.6	8.5	32.7	20.2	26.5	22.8	31.7	17.8	34.7	5.9	15.0	4.4
		20.33	*2.02*	*1263*			*6.8*	*5.7*	*5.1*	*1.1*	*2.0*	*1.1*	*4.9*	*20.3*	*7.3*	*43.7*	*19.4*	*24.4*	*28.9*	*19.6*	*17.3*	*28.2*	*4.9*	*15.5*	*5.0*
169	(2E)-oct-2-enal	30.36	1.78	1034	R	0.004	2.4	2.7	5.2	22.4	1.0	0.0	1.9	2.6	1.0	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.7	0.0
		27.25	*1.89*	*1424*			*3.0*	*2.9*	*6.1*	*20.9*	*1.0*	*0.0*	*1.7*	*2.6*	*0.8*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*0.0*
178	o-guaiacol	31.07	4.19	1049	Mo	0.02	5.3	0.0	28.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
185	(3E)-octa-3,5-dien-2-one	31.88	2.65	1065	V/Mo	0.0005	1.8	4.2	1.6	4.1	0.7	0.3	0.0	0.6	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.0	1.6	0.0	0.0
		30.91	*1.73*	*1561*			*2.3*	*4.6*	*1.9*	*3.8*	*0.7*	*0.4*	*0.0*	*0.6*	*0.0*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.3*	*0.0*	*0.0*
191	(3E)-6-methylhepta-3,5-dien-2-one	32.38	2.77	1076	V/Mo	0.38	4.6	4.4	1.9	1.9	0.0	0.0	0.6	0.6	0.5	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		33.83	*1.64*	*1582*			*5.8*	*4.8*	*2.2*	*1.8*	*0.0*	*0.0*	*0.5*	*0.6*	*0.5*	*0.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
196	nonanal	32.77	1.33	1084	R	0.15	110.8	64.4	137.2	56.4	11.7	3.4	31.5	34.6	15.9	13.9	8.2	10.4	2.8	5.8	7.0	9.4	33.2	7.4	2.3
		25.75	*2.19*	*1388*			*139.3*	*70.8*	*160.5*	*52.6*	*11.7*	*4.6*	*28.1*	*34.1*	*13.6*	*18.6*	*7.8*	*9.6*	*3.5*	*3.6*	*6.8*	*7.7*	*27.4*	*7.6*	*2.6*
213	ethyl cyclohexanecarboxylate	34.44	1.33	1119	Fr	0.00016	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
217	(2Z)-non-2-enal	35.44	1.57	1141	R	0.0045	0.0	0.0	0.0	0.0	2.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
218	(2E)-non-2-enal	35.45	1.75	1141	R	0.9	1.3	1.0	2.2	4.3	0.5	0.0	0.8	0.6	0.4	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.9	0.4	0.0
		31.75	*1.98*	*1531*			*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
230	ethyl octanoate	37.68	1.02	1189	V	10	6.7	2.8	0.4	0.0	0.0	0.0	0.0	0.5	0.6	0.0	2.9	0.0	0.8	0.0	0.0	0.4	0.0	0.0	0.0
		27.50	*2.36*	*1429*			*8.4*	*3.1*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.5*	*0.5*	*0.0*	*2.8*	*0.0*	*1.0*	*0.0*	*0.0*	*0.3*	*0.0*	*0.0*	*0.0*
235	Decanal	37.94	1.27	1195	R	0.65	1.6	1.3	2.8	1.7	0.4	0.0	0.3	1.0	0.5	0.4	0.0	0.0	0.0	0.0	0.0	0.0	2.0	1.2	0.0
		30.25	*2.23*	*1494*			*2.0*	*1.5*	*3.3*	*1.6*	*0.4*	*0.0*	*0.3*	*1.0*	*0.4*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.6*	*1.2*	*0.0*
245	(2E)-dec-2-enal	40.03	1.75	1243	R	0.01	7.6	3.6	7.8	7.6	1.0	0.4	2.2	6.6	2.8	1.3	1.4	3.2	1.7	0.8	0.6	3.1	2.4	2.3	0.9
		36.08	*2.02*	*1638*			*9.6*	*4.0*	*9.2*	*7.1*	*1.0*	*0.5*	*1.9*	*6.5*	*2.4*	*1.8*	*1.3*	*3.0*	*2.2*	*0.5*	*0.5*	*2.5*	*2.0*	*2.4*	*1.0*
249	(2E, 4Z)-deca-2,4-dienal	41.29	2.32	1273	R	0.01	0.3	0.0	0.5	1.2	2.3	0.0	0.0	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.0
		40.58	*1.73*	*1756*			*0.4*	*0.0*	*0.6*	*1.2*	*2.3*	*0.0*	*0.0*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.3*	*0.0*	*0.0*
251	(2E,4E)-deca-2,4-dienal	42.29	2.53	1296	R	0.18	0.3	0.0	0.4	1.7	3.6	0.0	0.4	0.6	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.3	0.0	0.0
		42.25	*1.64*	*1800*			*0.3*	*0.0*	*0.4*	*1.7*	*4.8*	*0.0*	*0.4*	*0.6*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*2.4*	*0.0*	*0.0*
256	ethyl decanoate	46.19	1.08	1393	V	10	1.0	1.1	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	1.6	0.5	1.6	2.4	3.7	1.1	0.7	0.5
		35.91	*2.48*	*1634*			*1.2*	*1.2*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.0*	*1.4*	*0.6*	*1.0*	*2.3*	*3.0*	*0.9*	*0.7*	*0.6*

Table 2. List of compounds adopted in the chemical blueprint of samples. ^{1}D and ^{2}D retention times, experimental I^{T}_{S}, known correlation with sensory defect, odor threshold values (OT in mg/kg) and normalized peak volume (multiplied per 10^{5}) are reported for both apolar × polar and polar × apolar (in *italic*) column set.

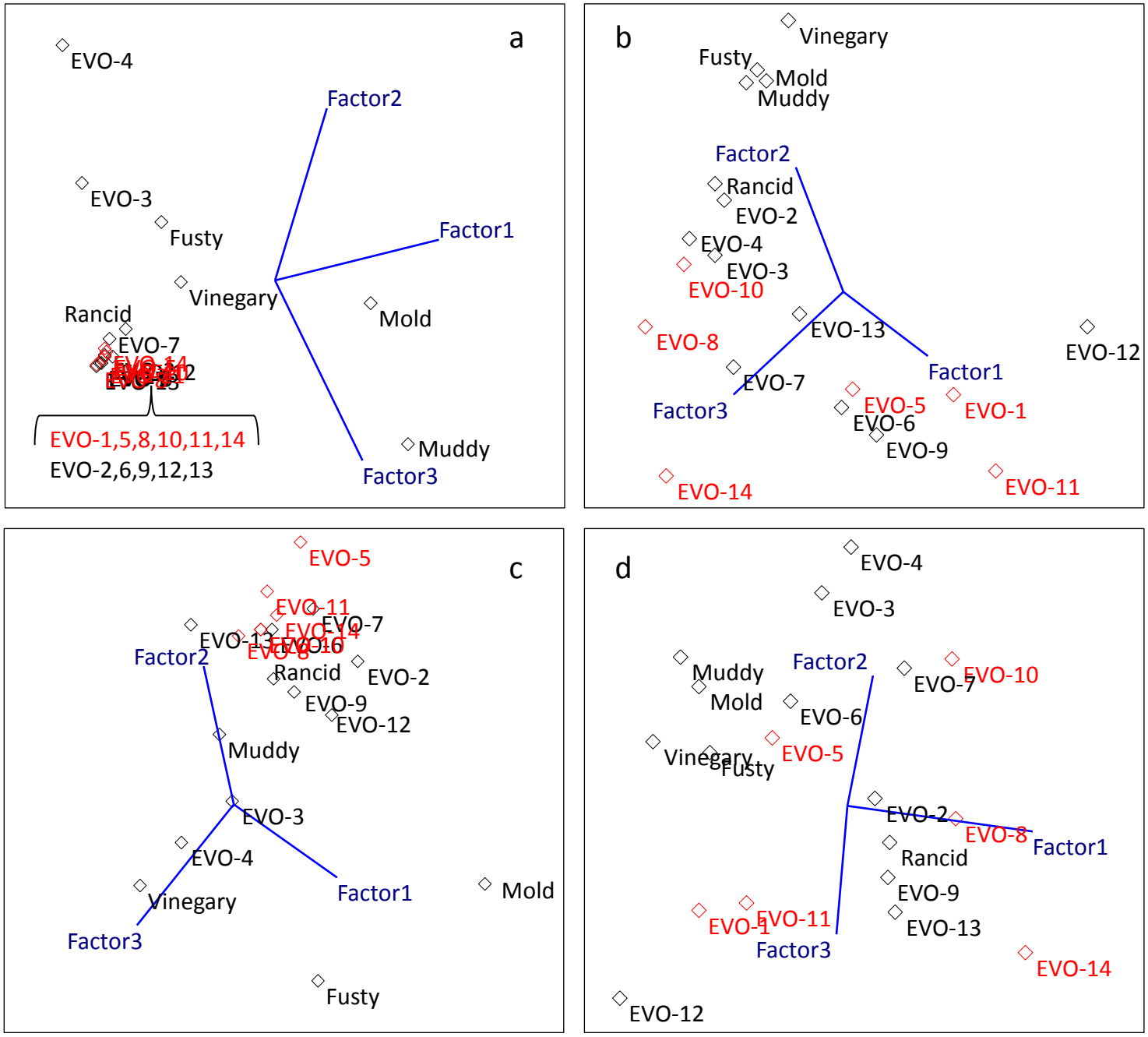
ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory defect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
1	ethanol	2.78	1.72	552	V	30	3.7	1.0	2.8	4.3	6.3	2.1	6.2	0.0	114.9	2.6	79.0	3.4	29.2	24.1	18.5	12.3	29.7	8.5	5.5
		7.67	*1.14*	*883*			*4.7*	*1.3*	*3.3*	*4.0*	*6.3*	*2.8*	*5.6*	*0.0*	*65.0*	*4.0*	*75.7*	*3.1*	*37.0*	*14.9*	*18.0*	*10.0*	*20.0*	*8.8*	*6.2*
6	butanal	6.85	1.05	607	F/M	0.018	11.3	0.0	0.3	2.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		7.00	*2.48*	*857*			*14.2*	*0.0*	*0.4*	*3.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
7	ethyl acetate	7.52	0.96	613	F/V	0.94	18.9	17.2	5.3	4.7	0.0	0.0	0.0	9.4	16.9	0.0	85.7	40.2	12.4	10.7	8.5	2.8	0.0	3.2	0.0
		6.75	*1.35*	*850*			*23.8*	*18.9*	*6.2*	*4.3*	*0.0*	*0.0*	*0.0*	*9.2*	*9.6*	*0.0*	*82.1*	*37.1*	*15.8*	*6.6*	*8.3*	*2.3*	*0.0*	*3.2*	*0.0*
9	acetic acid	8.11	1.66	622	F/V/R	0.5	12.5	23.7	18.4	32.9	12.6	4.6	12.6	21.0	194.2	5.6	3.6	37.0	13.5	5.0	2.9	6.7	0.7	1.1	8.1
		28.50	*0.97*	*1457*			*15.7*	*26.0*	*21.5*	*30.7*	*12.6*	*6.2*	*11.2*	*20.8*	*109.9*	*8.6*	*3.4*	*34.1*	*17.1*	*5.0*	*2.9*	*7.3*	*0.7*	*1.0*	*6.8*
12	butan-1-ol	9.47	2.92	648	V/M *	0.15	1.6	4.0	0.0	17.1	0.0	0.3	1.3	1.8	0.6	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.2	0.0
		14.42	*1.26*	*1120*			*1.5*	*3.6*	*0.0*	*16.0*	*0.0*	*0.4*	*1.1*	*1.8*	*0.4*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.2*	*0.0*
13	3-methylbutanal	9.52	0.96	649	F/V	0.013	0.0	1.2	0.0	0.1	0.0	0.0	0.0	0.6	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		8.75	*2.61*	*927*			*0.0*	*1.5*	*0.0*	*0.1*	*0.0*	*0.0*	*0.0*	*0.6*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
14	pent-1-en-3-one	10.61	1.39	668	M	0.00073	0.7	1.6	0.6	2.4	1.4	29.5	2.0	6.1	0.4	4.6	0.9	24.3	1.5	0.0	3.4	2.8	0.3	0.5	8.9
		10.17	*1.47*	*983*			*0.9*	*1.8*	*0.8*	*2.2*	*1.4*	*39.6*	*1.8*	*6.0*	*0.2*	*7.1*	*0.9*	*22.4*	*1.9*	*0.0*	*3.3*	*2.3*	*0.2*	*0.6*	*10.1*
15	pent-1-en-3-ol	10.71	2.77	670	F/Mo	0.4	1.6	6.8	1.2	11.3	11.6	18.0	8.4	7.5	9.6	15.1	6.0	8.3	9.4	11.7	11.8	10.8	7.4	5.3	4.5
		15.08	*1.30*	*1135*			*2.0*	*7.5*	*1.4*	*10.6*	*11.6*	*24.2*	*7.5*	*7.4*	*8.2*	*20.2*	*5.8*	*7.6*	*11.9*	*7.3*	*11.5*	*8.8*	*6.1*	*5.4*	*5.1*
16	pentan-2-one	10.77	1.18	671	V	70	0.5	1.8	0.5	0.9	0.0	0.0	0.5	1.0	0.0	1.8	0.5	0.0	12.3	23.8	0.0	0.0	0.4	1.9	0.8
		8.84	*1.47*	*930*			*0.7*	*1.9*	*0.6*	*0.8*	*0.0*	*0.0*	*0.4*	*0.9*	*0.0*	*2.4*	*0.5*	*0.0*	*15.6*	*14.8*	*0.0*	*0.0*	*0.3*	*1.9*	*0.9*
20	2-methylbutanal	11.02	1.15	676	V/M *	0.01	3.8	16.0	7.5	9.3	0.7	16.7	20.5	18.2	5.1	0.0	16.3	0.0	0.0	0.0	21.1	18.9	8.4	19.2	0.0
		4.34	*1.09*	*750*			*4.7*	*17.6*	*8.7*	*8.7*	*0.7*	*22.4*	*18.3*	*18.0*	*4.4*	*0.0*	*15.6*	*0.0*	*0.0*	*0.0*	*20.5*	*15.3*	*6.9*	*19.8*	*0.0*
23	pentan-3-ol	11.75	4.97	689	V	9	0.5	14.0	0.0	0.0	0.0	0.8	0.0	0.0	1.2	0.0	1.8	0.8	0.8	1.0	0.0	0.0	0.0	0.0	0.0
		13.00	*1.35*	*1078*			*0.6*	*11.0*	*0.0*	*0.0*	*0.0*	*1.1*	*0.0*	*0.0*	*1.0*	*0.0*	*1.7*	*0.7*	*1.0*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
25	ethyl propanoate	12.10	0.93	696	F	0.1	4.8	0.4	0.4	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
28	methyl butanoate	12.43	1.05	702	F	0.06	0.8	0.0	0.0	4.4	0.0	0.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
31	3-methylbutan-1-ol	13.46	2.62	720	F/M/Mo	0.1	2.4	8.7	1.0	2.9	0.0	1.0	5.3	18.1	23.1	3.1	15.5	11.5	4.9	8.0	3.3	4.1	2.8	3.6	1.2
		18.35	*0.94*	*1215*			*3.0*	*9.6*	*1.1*	*2.7*	*0.0*	*1.4*	*4.7*	*17.9*	*19.8*	*4.1*	*14.9*	*10.6*	*6.2*	*4.9*	*3.2*	*3.3*	*2.3*	*3.7*	*1.4*
32	(2E)-pent-2-enal	13.53	1.93	721	V	0.3	5.9	12.9	6.9	21.4	10.5	4.4	3.4	5.7	1.2	4.5	1.6	0.0	1.2	0.5	1.4	1.1	2.9	1.3	1.0
		14.08	*1.52*	*1110*			*7.4*	*14.2*	*8.1*	*20.0*	*10.5*	*6.0*	*3.0*	*5.6*	*1.0*	*6.0*	*1.5*	*0.0*	*1.5*	*0.3*	*1.4*	*0.9*	*2.4*	*1.3*	*1.1*
39	pentan-1-ol	14.88	2.92	746	F/M/V	3	1.0	2.1	0.9	4.5	1.9	1.0	3.5	2.1	2.0	3.7	4.0	5.8	1.0	6.0	1.3	1.4	3.4	7.4	0.0
		19.00	*1.35*	*1231*			*1.0*	*2.1*	*0.9*	*4.5*	*1.9*	*1.0*	*3.5*	*2.1*	*2.0*	*3.7*	*4.0*	*5.8*	*1.0*	*6.0*	*1.3*	*1.4*	*3.4*	*7.4*	*0.0*
45	(3Z)-hex-3-enal	15.86	1.66	764	Fr	0.003	0.0	3.2	1.3	4.7	11.8	14.2	7.6	2.7	0.0	5.3	1.5	1.7	3.4	0.7	1.8	3.9	28.9	15.8	9.4
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
46	Hexanal	15.94	1.33	773	F/Mo/V/R	0.08	92.8	170.7	209.1	7.6	59.8	20.6	37.1	44.7	14.7	22.2	15.8	16.3	25.1	32.9	29.4	33.4	212.8	37.0	13.0
		12.25	*1.77*	*1054*			*116.7*	*187.7*	*244.7*	*7.1*	*59.8*	*27.7*	*33.1*	*44.2*	*12.6*	*29.6*	*15.2*	*15.0*	*31.9*	*20.4*	*28.6*	*27.1*	*175.8*	*38.0*	*14.8*
48	ethyl butanoate	17.10	0.93	786	F	0.03	5.2	0.7	0.0	31.3	0.0	0.0	0.0	1.6	1.6	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.0
		10.59	*1.77*	*1000*			*6.6*	*0.8*	*0.0*	*29.2*	*0.0*	*0.0*	*0.0*	*1.6*	*1.4*	*0.0*	*0.0*	*0.0*	*0.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*

Table 3. continued.

ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory defect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
49	oct-1-ene	17.18	0.54	787	M	0.08	3.9	1.6	2.7	1.5	0.0	0.0	3.9	4.8	7.9	0.7	0.0	0.0	0.0	0.0	0.0	0.0	8.4	0.0	0.0
		6.09	*1.68*	*820*			*4.9*	*1.8*	*3.2*	*1.4*	*0.0*	*0.0*	*3.5*	*4.7*	*6.7*	*0.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*6.9*	*0.0*	*0.0*
52	butyl acetate	17.60	1.05	795	F	0.3	0.0	0.0	0.0	9.3	0.0	0.0	0.3	1.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		12.00	*1.73*	*1046*			*0.0*	*0.0*	*0.0*	*8.7*	*0.0*	*0.0*	*0.2*	*1.1*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
53	octane	17.68	0.51	800	F/V/R	0.94	51.9	115.2	65.3	75.4	0.4	0.9	21.9	80.2	51.1	9.3	24.1	31.8	8.6	7.2	7.5	12.2	20.4	42.7	0.0
		5.59	*1.89*	*800*			*65.3*	*126.7*	*76.4*	*70.3*	*0.4*	*1.2*	*19.5*	*79.2*	*43.8*	*12.4*	*23.1*	*29.3*	*10.9*	*4.5*	*7.3*	*9.9*	*16.9*	*43.9*	*0.0*
63	(2E)-hex-2-enal	19.11	1.84	822	Mo/V/F/R	0.42	1.0	1.7	1.9	8.2	565.4	582.0	396.0	37.0	10.4	209.1	218.4	209.0	545.4	444.5	565.9	425.7	406.8	391.3	782.0
		18.00	*1.64*	*1208*			*1.2*	*1.8*	*2.3*	*7.7*	*565.4*	*781.4*	*353.0*	*36.5*	*8.9*	*279.5*	*209.2*	*192.6*	*692.1*	*275.2*	*551.0*	*345.5*	*336.1*	*402.5*	*886.9*
64	(3E)-hex-3-en-1-ol	19.31	3.69	826	F/R/V	6	0.7	0.4	0.6	0.8	1.6	0.6	0.7	1.4	2.6	2.6	3.7	2.6	1.5	2.1	1.3	3.0	1.9	5.9	2.2
		24.08	*1.30*	*1350*			*0.9*	*0.5*	*0.6*	*0.7*	*1.6*	*0.8*	*0.6*	*1.3*	*2.2*	*3.5*	*3.6*	*2.4*	*2.0*	*1.3*	*1.3*	*2.4*	*1.6*	*6.1*	*2.5*
66	(3Z)-hex-3-en-1-ol	19.55	3.25	830	V/F	1.5	1.4	0.6	0.0	15.8	20.0	9.3	1.2	73.1	75.8	58.2	71.8	83.2	34.5	52.1	49.8	42.7	5.1	6.8	18.4
		24.92	*1.35*	*1369*			*1.8*	*0.6*	*0.0*	*14.7*	*20.0*	*12.5*	*1.1*	*72.1*	*64.9*	*77.8*	*68.8*	*76.7*	*43.8*	*32.2*	*48.5*	*34.6*	*4.2*	*7.0*	*20.8*
68	(2E)-hex-2-en-1-ol	19.89	3.58	837	V	5	0.9	3.9	0.0	0.0	49.0	55.8	115.9	9.8	26.1	80.3	61.0	87.9	54.9	81.7	33.4	65.3	14.8	194.5	19.1
		25.92	*1.26*	*1392*			*1.1*	*4.3*	*0.0*	*0.0*	*49.0*	*75.0*	*103.3*	*9.7*	*22.4*	*107.3*	*58.5*	*81.0*	*69.6*	*50.6*	*32.6*	*53.0*	*12.2*	*200.0*	*21.6*
74	hexan-1-ol	20.63	2.71	850	Fr	0.4	1.3	4.6	1.3	19.2	0.0	25.1	33.6	23.8	40.3	34.9	59.4	98.5	54.0	82.0	39.3	54.1	11.0	79.5	17.5
		18.17	*1.81*	*1212*			*1.7*	*5.0*	*1.5*	*17.9*	*0.0*	*33.7*	*30.0*	*23.5*	*34.6*	*46.6*	*56.9*	*90.8*	*68.6*	*50.8*	*38.2*	*43.9*	*9.0*	*81.8*	*19.8*
80	heptan-2-one	21.85	1.27	872	V	0.3	3.8	10.0	7.6	7.5	0.5	0.3	0.7	2.5	0.9	3.4	2.7	1.6	0.6	0.6	0.0	1.4	2.1	0.0	0.0
		16.42	*1.89*	*1169*			*4.8*	*10.9*	*8.9*	*7.0*	*0.5*	*0.4*	*0.6*	*2.5*	*0.8*	*4.5*	*2.6*	*1.5*	*0.8*	*0.4*	*0.0*	*1.1*	*1.7*	*0.0*	*0.0*
85	heptanal	22.35	1.24	881	R	0.5	10.8	6.9	16.7	7.3	1.6	0.3	3.5	5.6	1.6	1.1	2.5	0.0	0.0	0.0	0.0	0.9	8.0	2.0	0.0
		16.50	*1.89*	*1161*			*13.6*	*7.6*	*19.5*	*6.8*	*1.6*	*0.4*	*3.1*	*5.5*	*1.4*	*1.5*	*2.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*6.6*	*2.0*	*0.0*
86	ethyl pentanoate	22.68	0.96	887	M	0.0015	1.3	0.9	0.0	0.0	0.0	0.0	0.0	0.0	0.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.8	1.3	0.0
		23.08	*2.27*	*1327*			*1.7*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*1.3*	*0.0*
103	(2Z)-hept-2-enal	24.28	1.72	917	R	0.042	0.7	0.8	1.2	2.4	0.5	0.0	0.2	0.0	0.0	0.6	0.0	0.0	0.0	0.0	0.0	2.1	1.3	3.1	0.0
		22.58	*1.77*	*1315*			*0.8*	*0.9*	*1.4*	*2.2*	*0.5*	*0.0*	*0.2*	*0.0*	*0.0*	*0.8*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.7*	*1.0*	*3.1*	*0.0*
110	(2E)-hept-2-enal	24.78	1.87	926	Mo/R	0.005	14.8	14.0	39.6	2.8	12.0	2.3	9.9	12.3	4.6	4.8	3.6	3.8	0.9	3.5	2.4	0.0	21.1	0.0	0.4
		22.67	*1.81*	*1317*			*18.6*	*15.5*	*46.3*	*2.6*	*12.0*	*3.1*	*8.8*	*12.2*	*4.0*	*6.4*	*3.5*	*3.5*	*1.2*	*2.2*	*2.3*	*0.0*	*17.4*	*0.0*	*0.5*
122	(2Z,4E)-hepta-2,4-dienal	26.30	2.98	955	R/Mo/F	0.36	0.7	1.7	0.6	0.7	1.2	0.0	2.7	11.6	0.0	0.0	4.1	0.0	0.0	0.0	0.0	0.0	0.0	1.7	0.0
		28.58	*1.60*	*1455*			*0.9*	*1.9*	*0.8*	*0.7*	*1.5*	*0.0*	*2.4*	*11.5*	*0.0*	*0.0*	*4.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.8*	*0.0*
123	oct-1-en-3-one	26.44	1.42	958	Mo	0.01	13.5	11.0	37.8	27.1	9.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	14.4	0.0	0.0	0.0	0.0	0.0
		21.58	*1.89*	*1292*			*16.9*	*12.1*	*44.3*	*25.3*	*9.1*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*8.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
125	(2E,4Z)-hepta-2,4-dienal	26.63	2.77	961	R/Mo/F	10	6.6	15.7	8.3	24.4	25.7	4.2	0.3	0.6	2.5	4.0	2.6	4.3	1.0	4.3	4.3	2.9	5.5	0.0	0.0
		28.66	*3.24*	*1457*			*8.3*	*17.2*	*9.7*	*22.8*	*25.7*	*5.7*	*0.3*	*0.6*	*2.2*	*5.3*	*2.5*	*4.0*	*1.3*	*2.6*	*4.2*	*2.4*	*4.6*	*0.0*	*0.0*
126	6-methylhept-5-en-2-one	26.69	1.63	962	Mo/F/R	1	148.2	88.6	88.6	70.7	2.1	0.8	4.7	11.0	4.4	4.0	2.9	3.0	0.0	0.0	0.0	1.9	4.5	2.0	0.0
		23.17	*1.85*	*1329*			*186.3*	*97.5*	*103.7*	*66.0*	*2.1*	*1.1*	*4.2*	*10.8*	*3.7*	*5.3*	*2.8*	*2.8*	*0.0*	*0.0*	*0.0*	*1.5*	*3.7*	*2.1*	*0.0*
131	oct-1-en-3-ol	27.04	2.26	969	Mo	0.05	4.0	3.1	11.2	26.2	2.6	0.5	2.1	2.0	1.2	1.3	1.6	0.0	0.5	0.0	0.0	0.0	1.2	2.0	0.0
		27.83	*1.47*	*1437*			*0.6*	*0.3*	*1.0*	*1.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
134	octan-2-one	27.35	1.27	975	V	0.51	4.4	4.9	7.3	5.9	0.5	0.4	0.6	1.0	1.6	3.6	2.6	0.9	0.0	0.9	1.7	0.8	1.3	0.5	0.0
		20.83	*2.06*	*1274*			*0.0*	*0.0*	*0.1*	*0.2*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*

Table 3. continued.

ID#		^{1}D (min)	^{2}D (s)	I^{T}_{S}	Sensory défect	OT (mg/kg)	Muddy	Vinegary	Mould	Fusty	Rancid	EVO-01	EVO-02	EVO-03	EVO-04	EVO-05	EVO-06	EVO-07	EVO-08	EVO-09	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
135	(2E,4E)-hepta-2,4-dienal	27.38	2.98	976	R/Mo/F	0.36	3.3	6.7	3.1	12.8	16.3	1.0	1.0	3.6	0.8	0.0	0.0	2.1	0.0	1.4	2.6	1.6	3.4	1.6	0.0
		28.75	*1.73*	*1459*			*4.1*	*7.3*	*3.6*	*11.9*	*16.3*	*1.3*	*0.9*	*3.6*	*0.7*	*0.0*	*0.0*	*1.9*	*0.0*	*0.8*	*2.6*	*1.3*	*2.8*	*1.6*	*0.0*
137	6-methylhept-5-en-2-ol	27.70	2.23	982	Mo	2	1.0	0.6	1.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
138	octanal	27.85	1.27	984	Mo/R	0.32	5.2	8.0	13.1	12.9	1.2	0.3	0.0	79.6	1.1	58.8	54.1	60.6	48.1	80.9	10.7	63.9	4.8	2.7	0.0
		21.08	*2.02*	*1280*			*6.5*	*8.8*	*15.3*	*12.0*	*1.2*	*0.3*	*0.0*	*78.6*	*0.9*	*78.6*	*51.8*	*55.9*	*61.1*	*50.1*	*10.4*	*51.8*	*3.9*	*2.8*	*0.0*
140	octan-2-ol	27.94	1.63	986	Mo	0.1	0.7	0.0	3.5	1.3	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		25.50	*1.60*	*1383*			*0.8*	*0.0*	*4.1*	*1.2*	*0.0*	*0.0*	*0.0*	*0.0*	*0.9*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
149	hexyl acetate	28.52	1.08	997	Fr	1.04	5.4	5.2	4.4	1.1	2.0	0.8	5.5	20.6	8.5	32.7	20.2	26.5	22.8	31.7	17.8	34.7	5.9	15.0	4.4
		20.33	*2.02*	*1263*			*6.8*	*5.7*	*5.1*	*1.1*	*2.0*	*1.1*	*4.9*	*20.3*	*7.3*	*43.7*	*19.4*	*24.4*	*28.9*	*19.6*	*17.3*	*28.2*	*4.9*	*15.5*	*5.0*
169	(2E)-oct-2-enal	30.36	1.78	1034	R	0.004	2.4	2.7	5.2	22.4	1.0	0.0	1.9	2.6	1.0	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.7	0.0
		27.25	*1.89*	*1424*			*3.0*	*2.9*	*6.1*	*20.9*	*1.0*	*0.0*	*1.7*	*2.6*	*0.8*	*1.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.7*	*0.0*
178	o-guaiacol	31.07	4.19	1049	Mo	0.02	5.3	0.0	28.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
185	(3E)-octa-3,5-dien-2-one	31.88	2.65	1065	V/Mo	0.0005	1.8	4.2	1.6	4.1	0.7	0.3	0.0	0.6	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.0	1.6	0.0	0.0
		30.91	*1.73*	*1561*			*2.3*	*4.6*	*1.9*	*3.8*	*0.7*	*0.4*	*0.0*	*0.6*	*0.0*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.3*	*0.0*	*0.0*
191	(3E)-6-methylhepta-3,5-dien-2-one	32.38	2.77	1076	V/Mo	0.38	4.6	4.4	1.9	1.9	0.0	0.0	0.6	0.6	0.5	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		33.83	*1.64*	*1582*			*5.8*	*4.8*	*2.2*	*1.8*	*0.0*	*0.0*	*0.5*	*0.6*	*0.5*	*0.4*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
196	nonanal	32.77	1.33	1084	R	0.15	110.8	64.4	137.2	56.4	11.7	3.4	31.5	34.6	15.9	13.9	8.2	10.4	2.8	5.8	7.0	9.4	33.2	7.4	2.3
		25.75	*2.19*	*1388*			*139.3*	*70.8*	*160.5*	*52.6*	*11.7*	*4.6*	*28.1*	*34.1*	*13.6*	*18.6*	*7.8*	*9.6*	*3.5*	*3.6*	*6.8*	*7.7*	*27.4*	*7.6*	*2.6*
213	ethyl cyclohexanecarboxylate	34.44	1.33	1119	Fr	0.00016	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
217	(2Z)-non-2-enal	35.44	1.57	1141	R	0.0045	0.0	0.0	0.0	0.0	2.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
		nr					*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
218	(2E)-non-2-enal	35.45	1.75	1141	R	0.9	1.3	1.0	2.2	4.3	0.5	0.0	0.8	0.6	0.4	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.9	0.4	0.0
		31.75	*1.98*	*1531*			*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*
230	ethyl octanoate	37.68	1.02	1189	V	10	6.7	2.8	0.4	0.0	0.0	0.0	0.0	0.5	0.6	0.0	2.9	0.0	0.8	0.0	0.0	0.4	0.0	0.0	0.0
		27.50	*2.36*	*1429*			*8.4*	*3.1*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.5*	*0.5*	*0.0*	*2.8*	*0.0*	*1.0*	*0.0*	*0.0*	*0.3*	*0.0*	*0.0*	*0.0*
235	Decanal	37.94	1.27	1195	R	0.65	1.6	1.3	2.8	1.7	0.4	0.0	0.3	1.0	0.5	0.4	0.0	0.0	0.0	0.0	0.0	0.0	2.0	1.2	0.0
		30.25	*2.23*	*1494*			*2.0*	*1.5*	*3.3*	*1.6*	*0.4*	*0.0*	*0.3*	*1.0*	*0.4*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.6*	*1.2*	*0.0*
245	(2E)-dec-2-enal	40.03	1.75	1243	R	0.01	7.6	3.6	7.8	7.6	1.0	0.4	2.2	6.6	2.8	1.3	1.4	3.2	1.7	0.8	0.6	3.1	2.4	2.3	0.9
		36.08	*2.02*	*1638*			*9.6*	*4.0*	*9.2*	*7.1*	*1.0*	*0.5*	*1.9*	*6.5*	*2.4*	*1.8*	*1.3*	*3.0*	*2.2*	*0.5*	*0.5*	*2.5*	*2.0*	*2.4*	*1.0*
249	(2E, 4Z)-deca-2,4-dienal	41.29	2.32	1273	R	0.01	0.3	0.0	0.5	1.2	2.3	0.0	0.0	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.0
		40.58	*1.73*	*1756*			*0.4*	*0.0*	*0.6*	*1.2*	*2.3*	*0.0*	*0.0*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.3*	*0.0*	*0.0*
251	(2E,4E)-deca-2,4-dienal	42.29	2.53	1296	R	0.18	0.3	0.0	0.4	1.7	3.6	0.0	0.4	0.6	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.3	0.0	0.0
		42.25	*1.64*	*1800*			*0.3*	*0.0*	*0.4*	*1.7*	*4.8*	*0.0*	*0.4*	*0.6*	*0.6*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*2.4*	*0.0*	*0.0*
256	ethyl decanoate	46.19	1.08	1393	V	10	1.0	1.1	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	1.6	0.5	1.6	2.4	3.7	1.1	0.7	0.5
		35.91	*2.48*	*1634*			*1.2*	*1.2*	*0.5*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*0.0*	*1.0*	*1.4*	*0.6*	*1.0*	*2.3*	*3.0*	*0.9*	*0.7*	*0.6*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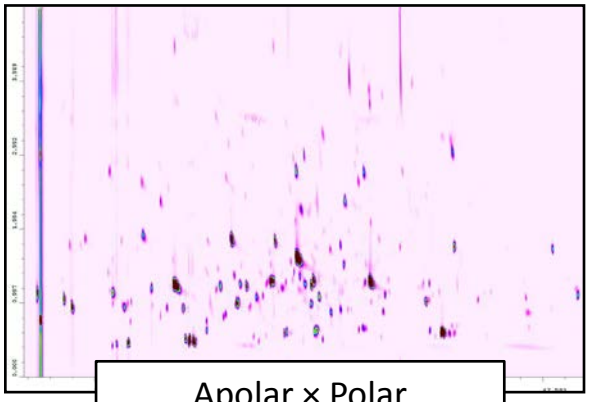
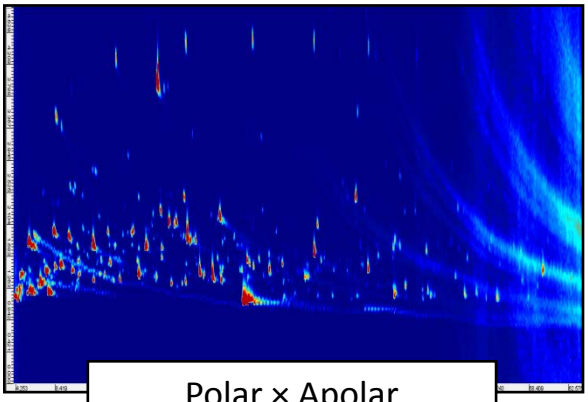
EVO-4
Factor2
EVO-3
Factor1
Fusty
Vinegary
Mold
Rancid
EVO-7
EVO-1,5,8,10,11,14
EVO-2,6,9,12,13
Muddy
Factor3
b
Vinegary
Fusty
Mold
Muddy
Factor2
Rancid
EVO-2
EVO-4
EVO-3
EVO-10
EVO-8
EVO-13
EVO-12
Factor1
EVO-7
Factor3
EVO-5
EVO-1
EVO-6
EVO-9
EVO-14
EVO-11
c
EVO-5
EVO-11
EVO-7
EVO-13
EVO-14
EVO-8
EVO-10
Factor2
Rancid
EVO-2
EVO-9
EVO-12
Muddy
EVO-3
EVO-4
Factor1
Mold
Vinegary
Factor3
Fusty
d
EVO-4
EVO-3
Muddy
Factor2
EVO-10
EVO-7
Mold
EVO-6
Vinegary
EVO-5
Fusty
EVO-2
EVO-8
Factor1
Rancid
EVO-9
EVO-1
EVO-11
EVO-13
Factor3
EVO-14
EVO-12



Apolar × Polar



Polar × Apolar

395 peak features

Untargeted analysis

452 peak features

Targeted analysis
Step 1

Identification LRI/EI-MS fragmentation

261 targets peaks

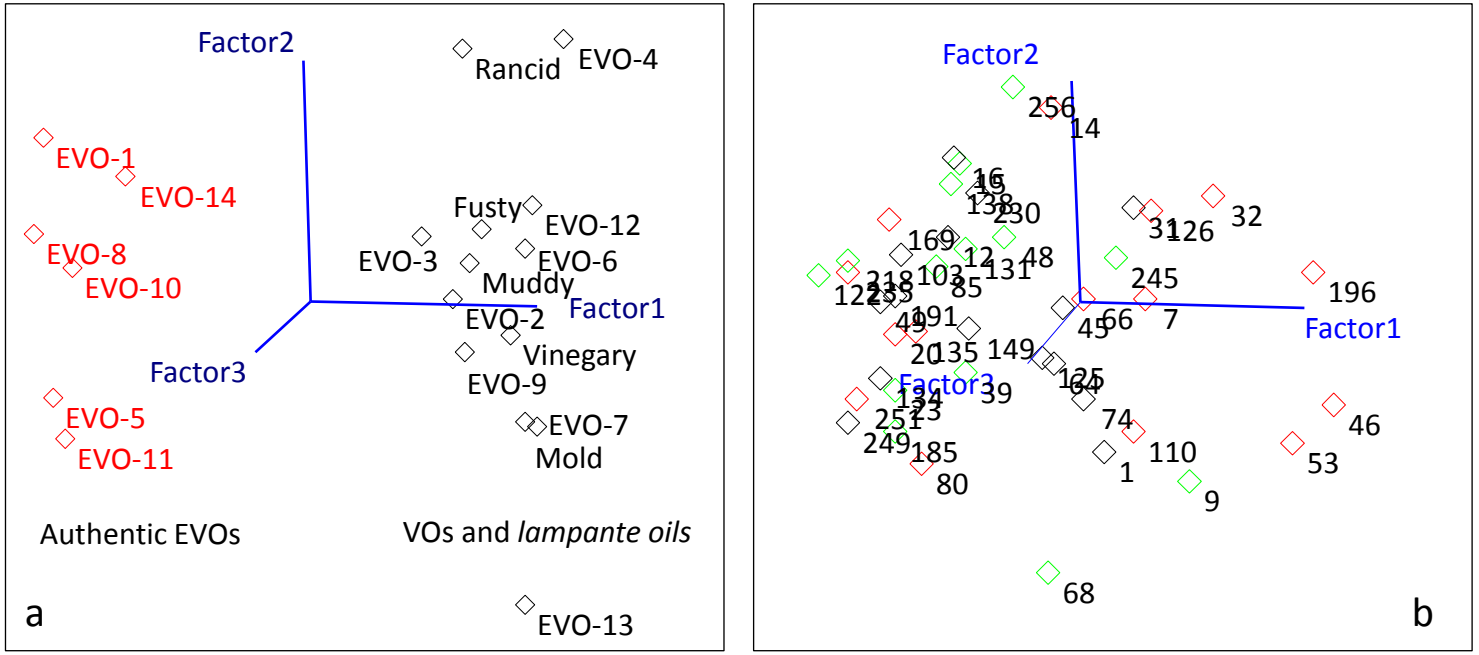
218 targets peaks

Pattern recognition
Chemica blueprint definition

Data reduction
Successive pair-wise comparisons to identify discriminant features

Targeted analysis
Step 2

Data reduction
Localization of most relevant analytes
OT-OAV


Factor2
Rancid
EVO-4
EVO-1
EVO-14
Fusty
EVO-12
EVO-8
EVO-3
EVO-6
EVO-10
Muddy
Factor1
EVO-2
Vinegary
Factor3
EVO-9
EVO-5
EVO-7
EVO-11
Mold
Authentic EVOs
VOs and lampante oils
a
EVO-13
Factor2
256
14
16
15
138
230
32
31
126
169
12
48
103
131
245
196
85
66
7
Factor1
191
45
135
149
Factor3
39
74
46
249
185
110
53
80
1
9
68
b

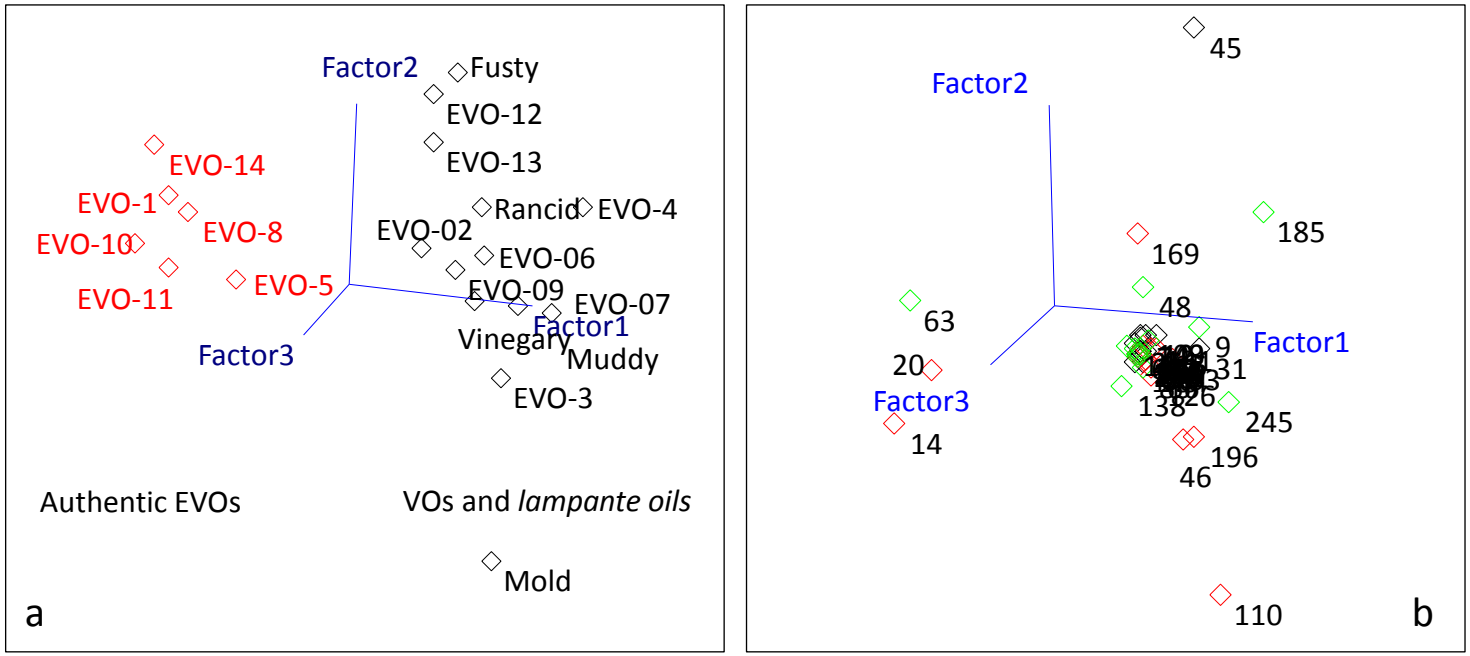

Factor2
Fusty
EVO-12
EVO-13
EVO-14
EVO-1
EVO-8
EVO-10
Rancid
EVO-4
EVO-02
EVO-06
EVO-11
EVO-5
EVO-09
EVO-07
Factor1
Vinegary
Factor3
Muddy
EVO-3
Authentic EVOs
VOs and *lampante oils*
Mold
a
45
Factor2
185
169
48
63
Factor1
9
20
31
Factor3
138
26
245
14
196
46
110
b

Table 2. List of identified compounds in the apolar × polar set, along with ^{1}D and ^{2}D retention times, experimental and library reported $I^{T}_{S,}$ known correlation with sensory defects, odor descriptors and MS similarity matching (%). Mo: *mould*; M: *muddy*; F: *fusty*; V: *vinegary*; R: *rancid*; Fr: *fruity*. Defects marked with * refer to correlation highlighted using the proposed iterative process.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^{T}_{S}	I^{T}_{S} lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
1	ethanol	2.78	1.72	552	482	V	Alcohol	95	92	94	96	93	92	94	95	94	94	96	96	97	97	98	98	96	98	97
2	tri-methylamine	4.60	1.15	560	577	Mo	fish	93	91	91	91				88											
3	ethyl ether	4.85	0.78	564	529		-				94															
4	Propan-1-ol	5.96	2.66	584	559	Mo	alcohol, pungent	92	90			91		90	88	96	96								95	
5	2-methylprop-2-enal	6.11	1.30	587	574	F	cooked, caramel	90	95					90	90											
6	butanal	6.85	1.05	607	607	F/M	pungent, green		93	96	94															
7	ethyl acetate	7.52	0.96	613	615	F/V/M	pineapple	91	90	91	91				91	92		90	88	96	95	93	94		91	
8	hexane	7.59	0.45	614	618		alkane								88	86										
9	acetic acid	8.11	1.66	622	622	F/V/R	sour, vinegary	96	92	91	92	90	91	90	92	90	91	90	96	96						
10	2-methyl-3-Buten-2-ol	8.28	1.78	626	600		herb	95	95	94	93	92		91	93		90								94	
11	2-Butenal	8.61	1.87	632	615		green, fruit	96	97	96	97	96	92	93	97		91									
12	butan-1-ol	9.47	2.92	648	662	V/M *	Winey	92	95		91		90	88	91	89	88								93	
13	3-methylbutanal	9.52	0.96	649	643	F/V	Malty	91	92						94	92										
14	pent-1-en-3-one	10.61	1.39	668	677	M	Mustard	95	95	95	95	96	94	95	95	90	95	87	90	98		98	98	98	98	98
15	pent-1-en-3-ol	10.71	2.77	670	671	F/Mo	wet earth	96	95	95	91	95	92	96	95	96	95	96	98	98	98	98	98	98	98	98
16	pentan-2-one	10.77	1.18	671	671	V *	ether	90	87	89	96			88	87		89	93		93	90			90	95	90
17	2-methylhexane	11.01	0.42	676	680		-				95						91									
18	methyl 2-methylpropanoate	11.02	0.87	676	680		flower			92																
19	2-Ethylfuran	11.02	0.99	676	689		sweet, ethereal	93	94	90	93				90		91									
20	2-methylbutanal	11.02	1.15	676	662	V/M *	Malty	89	95	90	95	90	91	90	94	92	90	91				92	90	92	90	
21	3,4-dihydro-2H-pyran	11.03	1.60	676	691	F/V *	-	90		90	91		90	89	91		88									
22	3-methylhexane	11.34	0.45	682	685		-	90			93															
23	pentan-3-ol	11.70	1.87	688	681	V	fruit	96			96		95		96	96		94	94	95	93					
24	2-butanol-3-one	11.75	4.97	689	716	V *	butter, cream											95	93							
25	ethyl propanoate	12.10	0.93	696	708	F	Fruit, strong	95	94	96	96															
26	propyl acetate	12.27	0.96	699	686	F *	sour	82			94															
27	heptane	12.34	0.45	700	700		alkane	95	94	90	96				96										93	
28	methyl butanoate	12.43	1.05	702	718	F	ether, fruit, sweet		95		95				88											
29	(2Z)-pent-2-enal	13.11	1.75	714	727	Mo	strawberry, fruit, tomato, green, pleasant	93	92		92	94	94	94	94		94									
30	(3Z)-pent-3-en-2-one	13.12	1.93	714	733		fish, pungent	94	93	95	91				90	90								90		90
31	3-methylbutan-1-ol	13.46	2.62	720	731	F/M/Mo	whiskey, malt, burnt	90	94	92	90		87	96	96	91	95	93	91	90	94	95	96	91	96	90
32	(2E)-pent-2-enal	13.53	1.93	721	751	V	green, apple, tomato, pungent	95	95	95	96	96	95	95	95	96	95	94		93	90	91	92	92	94	93
33	2-(chloromethyl)but-1-ene	14.02	0.96	730	711		-						86													
34	toluene	14.27	1.18	735	715		paint	89	97	90	96	93	95	97	95	95	97	94							85	
35	2-methylpentan-3-one	14.35	0.96	736	690		mint	80	91		90															
36	(2Z)-pent-2-en-1-ol	14.57	3.89	740	767		butter, pungent				89	95	93	90	91	90		90					80			90
37	(2E)-pent-2-en-1-ol	14.65	4.04	742	769		butter, pungent	88	90			97	96	97	96		96	94	90	91	95	90	90	90	91	91
38	ethyl 2-methylpropanoate	14.85	0.81	745	752		rancid, butter, cheese			95	92															
39	pentan-1-ol	14.88	2.92	746	752	F/M/V	Fruity	95	96	93	94	97	95	95	95	95	96	92	91	91	91	95	92	95	96	
40	(2E)-2-methylbut-2-enal	15.12	2.38	750	738	Mo *	green, fruit, aromatic	92	93	91	93	92	90	90	92	91	92							90		
41	methyl 3-methylbutanoate	15.35	0.99	754	721		sweat, acid, rancid			92																
42	2-methylpropyl acetate	15.43	0.93	756	768	F	fruit, apple, banana	91			93															
43	hexan-2-one	15.61	1.30	759	754		ether, grape			94						96	93									
44	2-methylpent-1-en-3-ol	15.71	2.59	761	747		alcohol, pungent	93	94		94	92	88		82	80										
45	(3Z)-hex-3-enal	15.86	1.66	764	797	Fr	green	92	95	90	87	95	95	95	96		96	92	90	93	90	91	92	93	93	93
46	Hexanal	15.94	1.33	773	801	F/Mo/V/R	green apple, grassy	95	96	96	97	97	96	96	96	98	97	95	95	96	96	96	95	90	96	95
47	1-methoxybutan-2-ol	15.99	4.64	766	756	R	-	91	91	90	91	92												92		
48	ethyl butanoate	17.10	0.93	786	803	F	sweet, fruity	95	85		96				96	97				90						
49	oct-1-ene	17.18	0.54	787	807	M	-	97	96	97	97			98	96	98	96							89		
50	propyl propanoate	17.43	0.93	792	785	Mo	pineapple				94															

Table 2. continued.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^T_S	I^T_S lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
51	(2Z)-oct-2-ene	17.51	0.42	793	824		-		92		91			97												
52	butyl acetate	17.60	1.05	795	785	F	green, fruity, pungent, sweet		95					91	90											
53	octane	17.68	0.51	796	800	F/V/R	alkane	95	91	90	88	91	96	92	88	86	96	96	90	94	96	96	96	91	91	
54	alkane isomer	17.68	0.48	796		*F/V/R* *	-																		90	
55	alkane isomer	17.93	0.48	801		*F/V/R* *	-																		90	
56	octa-1,3-diene	18.01	0.54	802	815		-	88				90		94										88		
57	(3Z)-hex-3-enal	18.03	1.63	803	850	Fr	green leaves, cut grass					93	92	93	91		92	92	90	95	92	93	93	90	95	95
58	(2Z)-oct-2-ene	18.34	0.54	809	824		-					91			95									94		
59	1-methoxyhexane	18.93	0.63	819	821		-	95	91	94	95				95	89		95	94	94		93	94			
60	1,1-dimethyl-2-(2-methylbut-3-en-2-yl)cyclopropane	18.93	0.75	819	859		-	87	87	87	87				88	88		92	92							
61	methyl 3-methylbut-2-enoate	18.94	1.45	819	770		-			94	93		94													
62	ethyl (2E)-but-2-enoate	19.02	1.39	821	839		-			89	95				94	90										
63	(2E)-hex-2-enal	19.11	1.84	822	850	Mo/V/F/R	Bitter almond, green	96	96	96	96	97	95	82	96	97	96	96	95	85	92	95	85	90	91	90
64	(3E)-hex-3-en-1-ol	19.31	3.69	826	830	F/R/V	green	95	96	92	93	95	93	88	95	95	95	93	93	90	94	85	88	90	96	90
65	3,4-dimethylhexan-2-one	19.35	0.99	827	824	Fr	-	86	86	87	89	90	86	87	88	90	87									
66	(3Z)-hex-3-en-1-ol	19.55	3.25	830	834	*V/F* *	green	92	91		94	96	96	95	95	96	95	96	96	96	96	93	96	90	96	96
67	ethylbenzene	19.85	1.18	836	857	Fr	strong	90	91	90	94		89	88	93	90		97							94	
68	(2E)-hex-2-en-1-ol	19.89	3.58	837	846	V	green grass, leaves	90			85	97	95	87	97	95	97	93	88	91	97	90	88	91	92	91
69	methyl (2Z)-2-methylbut-2-enoate	19.94	1.48	837	862	Fr	-		91	96	95				90	91										
70	1-ethenyl-3-methylidenecyclopent-1-ene	20.10	1.18	840	827		-			88			90	89			90									
71	1,3-cyclopentadiene	20.19	1.21	842	824		-	94	95	94	95	94	93	94	94	95	91	91	94	92			95		98	
72	ethyl 2-methylbutanoate	20.60	0.87	849	842	Fr	fruity			96	95				95	96		93	93							
73	ethyl 3-methylbutanoate	20.60	0.87	849	850	Fr	fruity	89		90	96			94												
74	hexan-1-ol	20.63	2.71	850	863	F	fruity, banana, soft	97	97	96	97	97	97	96	97	96	96	93	91	95	96	92	95	92	94	95
75	3-methylcyclopent-2-en-1-one	20.89	3.16	854	843	Fr	-	88			87		89													
76	(E)-3-ethoxy-2-methylprop-2-enal	20.95	2.26	856	867		-	90		89																
77	styrene	21.11	1.87	859	883		balsamic, gasoline	97	96	98	92		95	96	96	98	96							95		
78	p-xylene	21.36	1.36	863	877		plastic	96	94	95	97	96	96	96	95	96	97						92		90	
79	3-methylbutyl acetate	21.52	1.02	866	869		banana	96	87	92	95			93	94	87		92	90	91			90	91	92	
80	heptan-2-one	21.85	1.27	872	898	V	sweet, fruity	86	95	94	96	91	88	94	95	96	96	92	93	90	90		91	96		
81	ethyl 4-pentenoate	21.94	1.51	874	874	F	-	86	83	85	83	88		90	95											
82	(2E,4Z)-hexa-2,4-dienal	21.97	3.01	874	882		green					95	95	95	93		96	95		92	94		90	91	91	91
83	(2E,4E)-hexa-2,4-dienal	22.06	3.31	876	883		-	94	94	87	95	96	95	96	94		96	94	91	90		90	90	90	92	91
84	anisole	22.29	2.44	880	870		-		90	90	95				91	90										
85	heptanal	22.35	1.24	881	906	R	oily, fatty, woody	92	97	96	97	97	95	97	96	97	97	96					96	97	91	
86	ethyl pentanoate	22.68	0.96	887	884	M	-	92	96		94					93								85	87	
87	1-butoxybutane	22.96	2.83	892	892		-	87	90	88	90		88		89											
88	non-1-ene	23.01	0.54	893	906		-	90		91	95					96	86									
89	(2Z)-pent-2-en-1-yl acetate	23.11	1.30	895	892		-				90			91	95	92	96									
90	Nonane	23.43	0.48	900	900		alkane	90	91		96					96	88									
91	4-hydroxyhexan-3-one	23.43	1.02	901	916		-	88	90		91															
92	(2E)-hex-2-en-1-yl formate	23.44	1.51	901	922		-	90	92			96														
93	3-methylbut-2-en-1-yl acetate	23.52	1.42	902	901		-				95				90											
94	methyl hexanoate	23.69	1.11	905	922		fruit, fresh, sweet	93	93	89	86		87	90	93	95										
95	2,7-dimethyloxepine	23.69	1.39	905	916		-	90			90															
96	hexyl formate	23.94	1.24	910	929		-	95	96															94		
97	3-ethylocta-1,5-diene isomer	24.01	0.57	911	903		-				85														90	
98	cyclopentylmethanol	24.07	4.46	913	903		-	86			83	85		87	83		86									
99	3-ethylocta-1,5-diene isomer	24.09	0.45	913	949		-							92												
100	(3E)-non-3-ene	24.10	0.67	913	924		-									90										

Table 2. continued.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^{T}_{s}	I^{T}_{s} lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
101	ethyl (2E)-2-methylbut-2-enoate	24.11	1.33	913	929		-			95					94	96										
102	gamma-pentanolactone	24.12	1.93	913	941		-	92		84	88			87												
103	(2Z)-hept-2-enal	24.28	1.72	917	927	R	oxidized, tallowy	86	88	88	87	86		88			87						92	88	94	
104	(3E)-nona-1,3-diene	24.43	0.78	919	914	R *	-				88	90				94										
105	3-(propan-2-yl)cyclohex-1-ene	24.44	1.30	920	897		-	84	81	81	81	90	83	83	84	83	35							85		
106	2,6-dimethylocta-1,3,7-triene	24.44	1.51	920	916						83															
107	Benzaldehyde	24.66	4.49	924	952		almond, burnt sugar	96	94	96	90	92	92	97	92		96	93		93	95	92	94	93	92	92
108	3,4-dihydro-2H-pyran-2-carbaldehyde	24.68	0.93	924	940	Mo	-	88	90	90	91	90	92	90	91		93	92								88
109	methyl 2,4-dimethylpent-4-enoate	24.77	0.87	926	887		-				82															
110	(2E)-hept-2-enal	24.78	1.87	926	931	Mo/R	-	97	95	97	97	97	97	96	97	96	97	95	95	92	93	90		90		88
111	1-butylcyclopent-1-ene	25.18	0.64	932	969	R	-									96										
112	2,6-dimethylheptan-4-one	24.82	4.16	926~~.8~~	945		-																	88		
113	7-ethylcyclohepta-1,3,5-triene	25.19	1.15	934	946		-										91									
114	3-ethylocta-1,5-diene	25.59	0.60	942	949		-	95	90		96	96	96	94	94	94	84	95	90	90	90	94	90	90	90	90
115	cumene	25.77	1.30	945	928		-	90		88	90	88	93	90	89	90	91						90			
116	(2E)-hept-2-en-1-ol	25.81	3.40	946	964		-	91	96	93	92			94	92	96							90		94	
117	3-ethylocta-1,5-diene isomer	25.83	4.49	946	949		.	96		90	95	96	95	82	95	94	95	90		90	90	90		90	90	90
118	Phenol	25.99	4.67	949	901		Phenol				91								95							
119	3-ethylocta-1,5-diene isomer	26.09	0.60	951	949		-				92															
120	heptan-1-ol	26.13	2.53	952	960		herb	90	91	92	90			97	96	97							90	92	90	91
121	3,3-dimethyloctane	26.27	0.93	954	931						84															
122	(2Z,4E)-hepta-2,4-dienal	26.30	2.98	955	974	R/Mo/*F**	Fatty, rancid	84	85	84	90			86	88			88							90	
123	oct-1-en-3-one	26.44	1.42	958	972	Mo	mushroom, mould	95	95	95	95	92			94	93										
124	(methoxymethyl)benzene	26.54	2.11	959	969	V	-	90	93		94				93	95	91									
125	(2E,4Z)-hepta-2,4-dienal	26.63	2.77	961	971	R/Mo/F*	Fatty, rancid	93	93	93	93	92	93	93	92	92	92	90	91	90	92	95	91	91		
126	6-methylhept-5-en-2-one	26.69	1.63	962	981	Mo/F/R	pungent, green	92	93	85	86	94	94	94	94	94	94	93	91				92	95	91	
127	octan-3-one	26.94	1.18	967	952	M	herb, butter, resin	90	94	94	93		92	95	93	95								91	90	
128	1-ethyl-3-methylbenzene	26.94	1.36	967	967		-	90	91	88	93			90	93								90			
129	Sabinene	27.01	0.66	968	972		pepper, turpentine, wood	88	90	90	91		90	95	88	90								85		
130	1,3,5-trimethylbenzene	27.02	1.21	969	994		-	88			92		93	93		93	94								83	
131	oct-1-en-3-ol	27.04	2.26	969	969	Mo	mould, earthy	96	96	96	96	96	95	96	96	96		92		91				94	95	
132	hex-3-ene-2,5-diol	27.07	3.92	969~~.5~~	950	M	-																	85		
133	2-pentylfuran	27.35	1.02	975	984		green bean, butter	92	91	90	92	94														
134	octan-2-one	27.35	1.27	975	989	V	mould, green	96	96	95	95	91	92	93	95	96	96	95	95		93	90	94	92	93	
135	(2E,4E)-hepta-2,4-dienal	27.38	2.98	976	971	R/Mo/F*	Fatty, rancid	95	96	95	96	96	95	95	95	95	95		93		90	91	90	91	90	
136	1-methoxy-2-methylbenzene	27.45	1.99	977	983		-						94				90									
137	6-methylhept-5-en-2-ol	27.70	2.23	982	989	Mo	herbaceous, pungent	90		91	93															
138	octanal	27.85	1.27	984	998	Mo/R	fatty, sharp	97	97	96	97	97	97	97	95	94	95	95	91	93	85	96	90	97	95	
139	ethyl hexanoate	27.93	0.99	986	997	F	apple peel, fruit	95	92	91	95				94	93	92									
140	octan-2-ol	27.94	1.63	986	999	Mo	moss, nut, mushroom		91	95	92					91										
141	phenylmethanol	28.08	4.70	989	1026		-		86																	
142	3-ethylocta-1,5-diene isomer	28.09	0.51	989			-	95			92		94	94	94		92					90				90
143	(3Z)-hex-3-en-1-yl acetate	28.19	1.30	991	984		green, banana	94	95	94	91	96	94	96	92	95	94	93	91	95	95	95	91	96	95	96
144	3-ethylocta-1,5-diene isomer	28.26	0.66	992			-	82										89	90	91	90	90	90	90	90	
145	1,2,4-trimethylbenzene	28.28	1.54	992	1020		-										93									
146	dec-1-ene	28.34	0.57	994	1005		-									96										
147	3-ethylocta-1,5-diene isomer	28.34	0.63	994			-				93	94	94		94			90	90					90	90	90
148	dimethyl-2,5-dihydrofuran-2,5-dione	28.46	2.32	996	996		-	93	91		94					90	92									
149	hexyl acetate	28.52	1.08	997	984	Fr	green, fruity, sweet	95	95	95	96	96	95	96	95	94	94	97	96	97	94	97	95	96	97	96
150	(2E)-hex-2-en-1-yl acetate	28.52	1.18	997	993		-	90			91	85	91	85	88	90	93		91	94	93		90	90	93	

Table 2. continued.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^{T}_{s}	I^{T}_{s} lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
151	alpha-phellandrene	28.60	0.69	999	1007		dill				88			93	90	89	88									
152	4-hydroxyhex-2-enoic acid	28.72	2.95	1001	968		spice	90	87	88	87	90	88	91	90	89										
153	5-ethyl-2,5-dihydrofuran-2-one	28.79	2.20	1002	977		-			88		87														
154	decane	28.84	0.51	1003	1000		alkane		90	92	96	93		91	90	90										
155	2-ethylbutanal	28.97	3.25	1006	997		-	88	81	85	83	85		86	84		83									
156	alpha-terpinene	29.01	0.72	1007	1018		lemon							95												
157	(1E)-ethylidenecycloheptane	29.03	1.99	1007	1023		-	84	83	82	83	83		84												
158	(3E)-oct-3-en-2-one	29.45	1.72	1016	1036		nut, crushed bug	95	95	95	95	95	92	96	95	93	91	95						95	91	
159	limonene	29.52	0.87	1017	1025		citrus, mint	92	86	85	96	94		95	96	95	90	92	88	93			92	88		
160	gamma-hexalactone	29.60	0.78	1019	1042		-	95	87	88	90	89		88		89										
161	5-ethenyl-5-methyloxolan-2-one	29.64	3.49	1020	1035		-			87																
162	(2Z)-oct-2-enal	29.69	1.66	1021	1049		green leaf, walnut	89	90	89		90		88										90		
163	beta-phellandrene	29.85	0.75	1024	1025		mint, terpentine			89				92												
164	ethyl (2E)-hex-2-enoate	29.86	1.30	1024	1041		-	92		94																
165	methyl 2-oxohexanoate	30.27	1.18	1033	1020		-	87	85	86	88															
166	acetophenone	30.32	4.37	1035	1029		must, flower, almond	90	85	92	92			90	91									85	90	90
167	(Z)-beta-ocimene	30.35	0.93	1034	1044	Mo	citrus, herb, flower		87	94	93		96	95	96	96								93	95	90
168	butan-2-ylbenzene	30.35	1.18	1034	1028		-				85															
169	(2E)-oct-2-enal	30.36	1.78	1034	1049	R	green, nut, fat	97	97	97	97	97		97	97	97									95	
170	(E)-beta-ocimene	30.43	0.90	1036	1044	Mo	sweet, herb				94						96									
171	2,2,7,7-tetramethyloctane	30.59	0.43	1039	1045		-									93										
172	p-cymene	30.60	1.21	1039	1040		solvent, gasoline, citrus				82			95			93									
173	camphenilone	30.69	1.54	1041	1078		-	84	84	84	84		84	84	84	84										
174	gamma-terpinene	30.76	0.81	1043	1018		gasoline, turpentine							91		91										
175	methyl 2-ethylhexanoate	30.77	0.93	1043	1019		-	91																		
176	1-(cyclohex-1-en-1-yl)ethan-1-one	30.79	2.38	1043	1027		-	84	81	81	82	82	83	82	81		83						84	83		
177	1-chlorooctane	31.02	0.90	1048	1042		-	88	87	90	92			88	89											
178	o-guaiacol	31.07	4.19	1049	1090	Mo	smoke, sweet, medicine			95	94															
179	3-methylbutyl butanoate	31.27	0.93	1053	1057	F/Mo	-				94				91	90										
180	octan-1-ol	31.54	2.32	1058	1059		moss, nut, mushroom	91	89	90	90		92	98	96	96	97								95	
181	trans-dihydro-rose oxide	31.63	2.89	1060	1068		-	87	86	86	86			88		86										
182	cyclopenta[b]pyridine	31.80	3.07	1064	1040		-			86	86					85										
183	methyl benzoate	31.80	3.04	1064	1060		-	90			85	95	93	91	91	88	93	88	90	97	88	94	94	90		
184	o-cymene	31.86	1.30	1065	1042									90	88		90									
185	(3E)-octa-3,5-dien-2-one	31.88	2.65	1065	1068	*V/Mo* *	geranium-like	93	81	81	93	87	85		86		85							88		
186	meta-cymenene	32.02	1.45	1068	1082	Me	-							92												
187	2-(oxolan-3-yl)oxolane	32.04	2.29	1069	1079		-		87	86	86			87												
188	terpinolene	32.10	0.84	1070	1086		-							92												
189	undeca-1,10-diene	32.18	0.64	1071	1095		-									87										
190	1-ethenyl-3-ethylbenzene	32.19	1.66	1072	1096		-	93	91	90			90	88	90	88										
191	(3E)-6-methylhepta-3,5-dien-2-one	32.38	2.77	1076	1102	*V/Mo* *	-	93	93	93	94			91	88	90	91									
192	nonan-2-one	32.44	1.27	1077	1089		hot milk, soap, green	96	95	96	96			94	96	91	92							90		
193	cis-sabinene hydrate	32.44	1.42	1077	1069		balsamic							93												
194	(4E)-non-4-enal	32.44	1.54	1077	1112		-	90	88	90	91				90	88										
195	pinocamphone	32.59	0.54	1080	1109		-							80												
196	nonanal	32.77	1.33	1084	1107	R	fatty, waxy, pungent	89	91	95	97	98	91	97	97	98	98	96	90	95	96	92	96	92	96	94
197	p-ethyl-anisole	32.95	2.08	1087	1082		-			92	90					88										
198	Phenethyl alcohol	32.97	3.13	1088	1113		-	90	91	90	91	94	90	92	91	88	96	90	90		91	90	90		90	
199	(3Z)-4,8-dimethylnona-1,3,7-triene	33.26	0.72	1094	1113		-	88	90			91		94	88		90	91			90					
200	hexyl propanoate	33.27	1.02	1094	1106		-	90		94																

Table 2. continued.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^T_s	I^T_s lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
201	undec-1-ene	33.34	0.48	1095	1105		-							97		97										
202	2-ethenyl-1,1-dimethyl-3-methylidenecyclohexane	33.35	0.84	1095	1071		-										85									
203	heptyl acetate	33.60	1.08	1100	1114		-			95							90									
204	linalool	33.70	1.84	1103	1101		flower, lavender				94				96	90	91							95		
205	undecane	33.84	0.54	1106	1100		alkane				96			90												
206	(2Z)-undec-2-ene	33.93	0.57	1108	1123		-									94										
207	allo-ocimene	33.94	1.08	1108	1128		-				90		91	90	92	90	90									
208	1,2-dimethoxybenzene	33.98	3.80	1109	1147		-			93	93															
209	(3E)-4,8-dimethylnona-1,3,7-triene	34.02	0.90	1109	1113		-	93	95	93	95		95	90	95		91		91	93			94	92	90	91
210	p-Mentha-1,3,8-triene	34.02	1.27	1110	1108		-						90	91	91	90										
211	methyl octanoate	34.19	1.11	1113	1123		orange	94	90	91	92				90	88										
212	(2E)-undec-2-ene	34.34	0.60	1117	1123		-									95										
213	ethyl cyclohexanecarboxylate	34.44	1.33	1119	1147	Fr	aromatic, fruity				93															
214	neo-allo-ocimene	34.60	1.15	1122	1140		-				88		90	91	91	91	91								91	
215	(3E)-non-3-en-2-one	34.61	1.51	1122	1137		-					95														
216	(4E)-undeca-1,4-diene	34.68	0.75	1124	1113		-									91										
217	(2Z)-non-2-enal	35.44	1.57	1141	1144	R	green, fatty					96														
218	(2E)-non-2-enal	35.45	1.75	1141	1163	R	paper-like, fatty	96	97	97	97	94		97	95	92	94							91	90	
219	4-ethylphenol	35.48	3.92	1141	1141		-	93	90	93	95				94											
220	2-ethylphenol	35.65	3.90	1145	1148		-	90	93	93	92					93										
221	ethyl benzoate	35.71	2.77	1146	1160		camomile, flower, celery	80		88	88	90	93		92	90	91									
222	(2E,9E)-undeca-2,9-diene	36.01	0.82	1153	1131		-									86										
223	2-methoxy-4-methylphenol	36.29	2.41	1159	1188		-				91															
224	methyl 2-hydroxybenzoate	36.57	3.95	1165	1190		-	92			92	96	94	93	91	94	94	90		91		92	91	93	90	92
225	(2Z)-non-2-en-1-ol	37.02	1.21	1175	1162		-	85	82																	
226	(2E)-non-2-en-1-ol	37.35	1.21	1182	1163		-		82		81															
227	decan-2-one	37.44	1.27	1184	1196		-	95		90	90				88	90										
228	hexyl butanoate	37.52	0.87	1186	1195		apple peel							82										83		
229	(Z)-linalool oxide	37.52	1.05	1186	1164		flower	85	81			83														
230	ethyl octanoate	37.68	1.02	1189	1196	*V**	fruit, fat	96		93	96				90	92		88		90			90			
231	alpha-terpineol	37.71	2.41	1190	1186		oil, anise, mint			94	93															
232	(2E,4E)-nona-2,4-dienal	37.71	2.59	1190	1210	R	watermelon		92	95	93	90		88												
233	dodec-1-ene	37.93	0.60	1195	1191		-	97	91	94	97			90		97										
234	(E)-linalool oxide	37.93	1.05	1195	1164		flower	81	80	81	80	80	81	88		80	85									
235	Decanal	37.94	1.27	1195	1201	R	penetrating, sweet, waxy	96	92	97	96	96		92	94	92	92							93	95	
236	octyl acetate	38.19	1.11	1200	1211		fruit			94	91															
237	Dodecane	38.34	0.57	1204	1200		alkane	93	97		91		91	93		97	90									
238	(2Z)-dodec-2-ene	39.18	0.60	1223	1194		-	91		93	92	92	92	92	92	93	91	93	92			90	91	90	90	90
239	(8E)-dodeca-1,8-diene	39.18	0.78	1223	1212		-							86		87										
240	alkene	39.68	0.57	1235			-	92	92	92	93		91		93	92	92			90						
241	2-phenylethyl acetate	39.80	3.16	1238	1254		-	95	93								90									
242	dodeca-1,11-diene	39.93	0.69	1241	1194		-	91									90									
243	Menth-1en-7-al	39.97	3.01	1242	1196		-	80		80	80				81	80										
244	alkene	40.01	0.57	1243			-	91		91					92											
245	(2E)-dec-2-enal	40.03	1.75	1243	1265	R	painty, fishy, fatty	97	97	96	97	97	97	97	97	97	96	95	94	95	93	90	92	96	92	94
246	tridec-1-ene	40.09	0.60	1245	1290		-									94	92									
247	alkene	40.09	0.60	1245			-				91					90										
248	4-ethylguaiacol	40.27	1.21	1249	1275		spice, clove	94	90	95	94															
249	(2E, 4Z)-deca-2,4-dienal	41.29	2.32	1273	1292	R	deep-fried		94	93	94	95			90									94		
250	ethyl nonanoate	42.10	1.05	1292	1297		-	92			93															

Table 2. continued.

ID#	Compound	^{1}D (min)	^{2}D (s)	I^{T}_{s}	I^{T}_{s} lib	Sensory defect	sensory descriptor	Vinegary	Fusty	Mould	Muddy	Rancids	EVO-1	EVO-2	EVO-3	EVO-4	EVO-5	EVO-6	EVO-7	EVO-8	EVO-9	EVO-10	EVO-11	EVO-12	EVO-13	EVO-14
251	(2E,4E)-deca-2,4-dienal	42.29	2.53	1296	1315	R	deep-fried		96	94	95	96		91	90	91								92		
252	4-ethyl-1,2-dimethoxybenzene	42.30	2.98	1296	1271		-			93	92															
253	methyl 2-methoxybenzoate	42.84	0.48	1310	1334		warm, flower, walnut						90		93	91										
254	(2E)-2-undecen-1-ol	43.91	4.70	1336	1365		-	81	82	84	82															
255	gamma-nonalactone	44.16	4.58	1343	1358		coconut, peach	94		91	90															
256	ethyl decanoate	46.19	1.08	1393	1395	*V**	grape	94		90	94							91	90	91	92	92	93	91	91	90
257	copaene	46.35	0.87	1397	1374		wood, spice	94	95	93	95	95	92	92	93	95	95	90		90		91	92	91	90	
258	tetradec-1-ene	46.43	0.66	1399	1403		-		91	90	93			90		90	91									
259	tetradecane	46.76	0.60	1408	1400		alkane		96		94				90		90									
260	geranyl acetone	48.45	1.72	1451	1453		magnolia, green	93	93	94	94				90		91									
261	alpha-farnesene	50.44	1.18	1502	1505		wood, sweet	94	91	92	90	95	90	90	90	96	95	90	91	90		90	91	90	92	93

a
Muddy
Vinegary
Mould
Fusty
Rancid
EVO-1
EVO-2
EVO-3
EVO-4
EVO-5
EVO-6
EVO-7
EVO-8
EVO-9
EVO-10
EVO-11
EVO-12
EVO-13
EVO-14
b
Muddy
Vinegary
Mould
Fusty
Rancid
EVO-1
EVO-2
EVO-3
EVO-4
EVO-5
EVO-6
EVO-7
EVO-8
EVO-9
EVO-10
EVO-11
EVO-12
EVO-13
EVO-14
relative
-3
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3